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NAVY DEPARTMENT
OFFICE OF NAVAL RESEARCH
WASHINGTON, D. C.

15 September 1953

Report No. 737

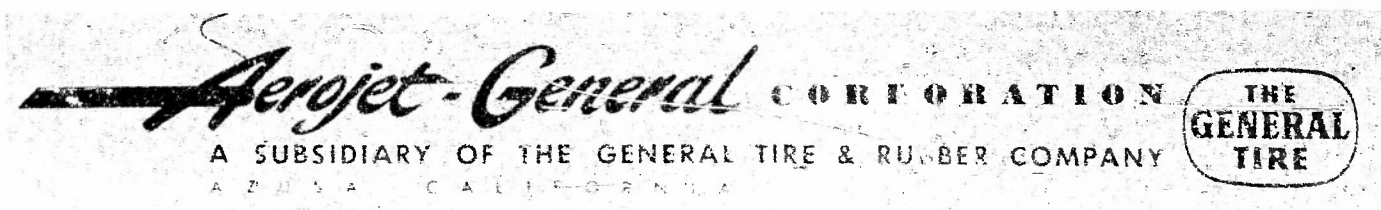
(Quarterly)

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EXPLOSIVES RESEARCH



Contract N7onr-46208



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15 September 1953

Report No. 737
(Quarterly)

EXPLOSIVES RESEARCH

Contract N7onr-46208

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AEROJET-GENERAL CORPORATION

Azusa, California

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1,3-bis(3',3',3'-Trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine

1,3,5-tris(3',3',3'-Trinitropropyl)-hexahydro-1,3,5-triazine

2,2,5,7,7,9,12,12-Octanitro-5,9-diaza-tridecane

2,2,4,7,7,10,12,12-Octanitro-4,10-diaza-tridecane

N,N'-bis(3,3,3-Trinitropropyl) oxamide

N,N'-Dinitro-N,N'-bis(trinitropropyl) oxamide

1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane

1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-5,7-diaza-decane

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CONTRACT FULFILLMENT STATEMENT

This quarterly report is submitted in partial fulfillment of
Contract N7onr-46209.

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I. SUMMARY

A. This quarterly report is submitted under Contract N7onr-46208 and covers the period 1 May through 31 July 1953.* The objectives of the contract are threefold:

1. Synthesis and physical studies of new high-energy polynitro compounds as potential explosives.
2. Desensitization of RDX with materials that will not lower the oxygen balance appreciably.
3. Development of bomb and shell casings made of an explosive plastic.

B. The more important results and conclusions are presented below:

1. Seven polynitrocarbamates were synthesized:
 - a. 2,2,9,9-Tetranitro-4-oxa-5-keto-6-aza-decane (I) from 3,3-dinitrobutyl isocyanate and 2,2-dinitropropanol.
 - b. 1,1,1,8,8-Pentanitro-3-oxa-4-keto-5-aza-nonane (II) from 3,3-dinitrobutyl isocyanate and 2,2,2-trinitroethanol.
 - c. 2,2,9,9,9-Pentanitro-4-oxa-5-keto-6-aza-nonane (III) from 3,3,3-trinitropropyl isocyanate and 2,2-dinitropropanol.
 - d. 1,1,1,8,8,15,15,15-Octanitro-4,12-diaza-5,11-diketo-6,10-dioxa-pentadecane (IV) from 3,3,3-trinitropropyl isocyanate and 2,2-dinitro-1,3-propanediol.
 - e. 1,1,1,8,15,15,15-Heptanitro-3,13-dioxa-4,12-diketo-5,8,11-triaza-pentadecane (V) from 3-nitrazo-1,5-pentane diisocyanate and 2,2,2-trinitroethanol.
 - f. 1,1,1,8,8,10,12,12,19,19,19-Undecanitro-3,17-dioxa-4,16-diketo-5,10,15-triaza-nonadecane (VI) from 3,3,5,7,7-pentanitro-5-aza-1,9-nonane diisocyanate and 2,2,2-trinitroethanol.
 - g. 1,1,1-Trinitro-3-oxa-4-keto-5-aza-hexane (VII) from methyl isocyanate and 2,2,2-trinitroethanol.

* Previous work on this contract was covered in Aerojet Reports No. 512, 536, 562, 589, 621, 637, 660, 682 and 711.

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I Summary, B (cont.)

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2. Seven postnitrated polynitrocarbamates were prepared:

- a. 2,2,6,9,9-Pentanitro-4-oxa-5-keto-6-aza-decane (VIII)
by the nitration of I.
- b. 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane (IX)
by the nitration of II.
- c. 2,2,6,9,9,9-Hexanitro-4-oxa-5-keto-6-aza-nonane (X)
by the nitration of III.
- d. 1,1,1,4,8,8,12,15,15,15-Decanitro-4,12-diaza-5,11-diketo-6,10-dioxa-pentadecane (XI) by the nitration of IV.
- e. 1,1,1,5,8,11,15,15,15-Nonanitro-3,13-dioxa-4,12-diketo-5,8,11-triaza-pentadecane (XII) by the nitration of V.
- f. 1,1,1,5,8,8,10,12,12,15,19,19,19-Tridecanitro-3,17-dioxa-4,16-diketo-5,10,15-triaza-nonadecane (XIII) by the nitration of VI.
- g. 1,1,1,5-Tetranitro-3-oxa-4-keto-5-aza-hexane (XIV) by the nitration of VII.

3. Four aliphatic polynitronitrates were prepared by the nitration of the corresponding alcohols:

- a. 4,4-Dinitro-1,7-heptanedinitrate (XV)
- b. 4,4,6,8,8-Pentanitro-1,11-undecanedinitrate (XVI)
- c. 4,4,6,6,8,8-Hexanitro-1,11-undecanedinitrate (XVII)
- d. 4,4,6,8,8-Pentanitro-6-aza-1,11-undecanedinitrate (XVIII).

4. Six polynitroamine salts were prepared by treating the corresponding isocyanates with dilute nitric or hydrochloric acid.

- a. 3,3-Dinitrobutyl ammonium nitrate (XIX)
- b. 3,3-Dinitro-1,5-pentane diammonium dinitrate (XX)
- c. 3-Nitrazo-1,5-pentane diammonium dinitrate (XXI)
- d. 3,6-Dinitraza-1,8-octane diammonium dinitrate (XXII)

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I Summary, B (cont.)

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e. 3,3,5,7,7-Pentanitro-5-aza-1,9-nonane diammonium dinitrate (XXIII)

f. 3,3,5,7,7-Pentanitro-5-aza-1,9-nonane diammonium dihydrochloride (XXIV).

5. The following samples were prepared and submitted to the Naval Ordnance Laboratory for evaluation as potential explosives:

- a. 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane
- b. 1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-5,7-diazadecane
- c. 4,4,6,8,8-Pentanitro-1,11-undecanedinitrate
- d. 4,4,6,6,8,8-Hexanitro-1,11-undecanedinitrate
- e. 4,4,6,8,8-Pentanitro-6-aza-1,11-undecanedinitrate
- f. 2,2,6,9,9-Pentanitro-4-oxa-5-keto-6-aza-decane
- g. 2,2,6,9,9,9-Hexanitro-4-oxa-5-keto-6-aza-nonane
- h. 1,1,1,4,8,8,12,15,15,15-Decanitro-4,12-diazas,11-diketo-6,10-dioxa-pentadecane
- i. 1,1,1,4,13,16,16,16-Octanitro-4,13-diazas,12-diketo-6,11-dioxa-8-hexadecyne
- j. 1,1,1,5,8,11,15,15,15-Nonanitro-3,13-dioxas,12-diketo-5,8,11-triasa-pentadecane
- k. 1,1,1,5,8,8,10,12,12,15,19,19,19-Tridecanitro-3,17-dioxa-4,16-diketo-5,10,15-triasa-nonadecane
- l. 3,3-Dinitrobutyl ammonium nitrate
- m. 3,3-Dinitro-1,5-pentane diammonium dinitrate
- n. 3-Nitrasa-1,5-pentane diammonium dinitrate
- o. 3,6-Dinitrasa-1,8-octane diammonium dinitrate
- p. 3,3,5,7,7-Pentanitro-5-aza-1,9-nonane diammonium dinitrate.

The preliminary evaluation of some of these compounds is described.

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I Summary, B (cont.)

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6. SPIA data sheets have been completed on the following compounds:

- a. 1,3-bis(3',3',3'-Trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine
- b. 1,3,5-tris(3',3',3'-Trinitropropyl)-hexahydro-1,3,5-triazine
- c. 2,2,5,7,7,9,12,12-Octanitro-5,9-diaza-tridecane
- d. 2,2,4,7,7,10,12,12-Octanitro-4,10-diaza-tridecane
- e. N,N'-bis(3,3,3-Trinitropropyl) oxamide
- f. N,N'-Dinitro-N,N'-bis(3,3,3-trinitropropyl) oxamide
- g. 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane
- h. 1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-5,7-diaza-decane.

II. TECHNICAL PROGRESS: SYNTHESIS OF NEW HIGH EXPLOSIVES

A. INTRODUCTION

1. The present explosives program is directed toward the synthesis of new, stable, high-energy polynitro compounds with a preferred oxygen balance on the positive side or very near zero on the negative side.

2. This report describes the synthesis of aliphatic polynitro-carbamates, polynitronitrates, and polynitroamine salts.

B. PREPARATION OF ALIPHATIC POLYNITROCARBAMATES

1. Discussion

a. The preparation of aliphatic polynitrocarbamates for evaluation as high explosives has been previously reported.* This work has now been extended utilizing the new isocyanates and alcohols prepared on the nitropolymer program. The following aliphatic polynitrocarbamates were synthesized.

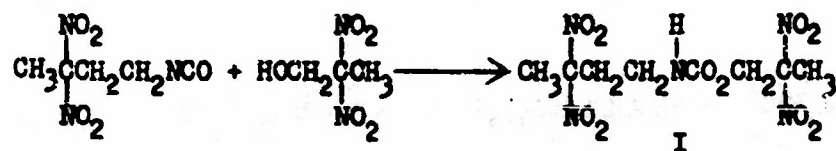
*Aerogel Reports No. 562, 589, and 621.

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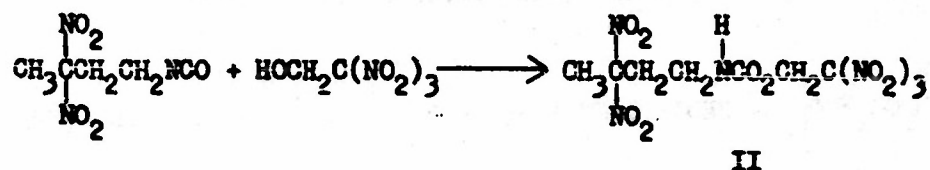
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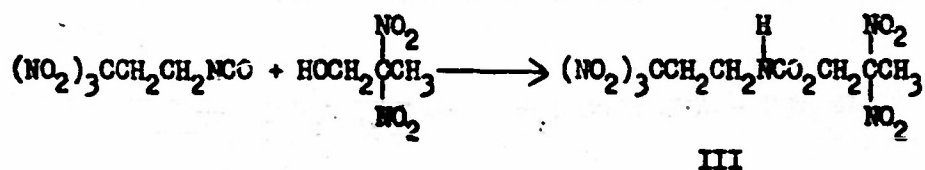
(1) 2,2,9,9-Tetranitro-4-oxa-5-keto-6-aza-decane
(I) from 3,3-dinitrobutyl isocyanate and 2,2-dinitropropanol:



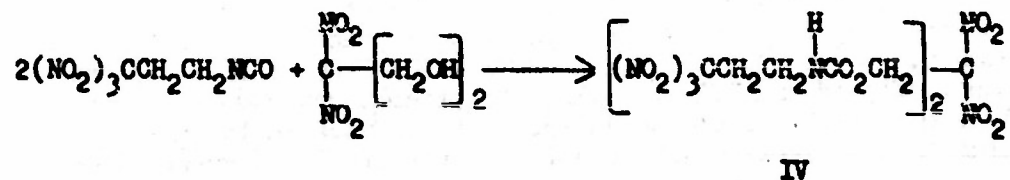
(2) 1,1,1,8,8-Pentanitro-3-oxa-4-keto-5-aza-nonane (II) from 3,3-dinitrobutyl isocyanate and 2,2,2-trinitroethanol:



(3) 2,2,9,9,9-Pentanitro-4-oxa-5-keto-6-aza-nonane (III) from 3,3,3-trinitropropyl isocyanate and 2,2-dinitropropanol:



(4) 1,1,1,8,8,15,15,15-Octanitro-4,12-diaza-5,11-diketo-6,10-dioxo-pentadecane (IV) from 3,3,3-trinitropropyl isocyanate and 2,2-dinitro-1,3-propanediol:

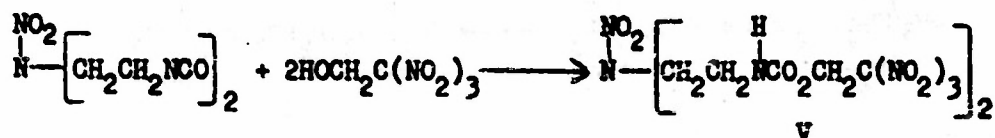


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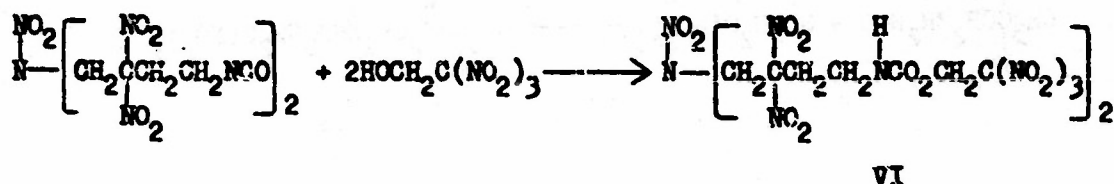
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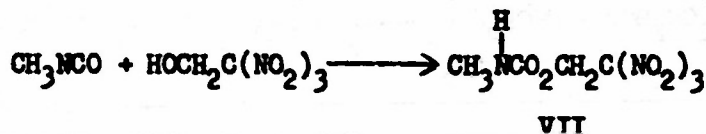
(5) 1,1,1,8,15,15,15-Heptanitro-3,13-dioxo-4,12-diketo-5,8,11-triaza-pentadecane (V) from 3-nitroso-1,5-pentane diisocyanate and 2,2,2-trinitroethanol:



(6) 1,1,1,8,8,10,12,12,19,19,19-Undecanitro-3,17-dioxo-4,16-diketo-5,10,15-triaza-nonadecane (VI) from 3,3,5,7,7-pentanitro-5-aza-1,9-nonane diisocyanate and 2,2,2-trinitroethanol:



(7) 1,1,1-Trinitro-3-oxo-4-keto-5-aza-hexane (VII) from methyl isocyanate* and 2,2,2-trinitroethanol:



2. Experimental

The general procedure employed was to reflux for about 8 hr equivalent quantities of the isocyanate and alcohol in dry, alcohol-free chloroform with a catalytic amount of ferric acetylacetonate. The solution was then evaporated to dryness in vacuo leaving a quantitative yield of product. Compounds III, V, and VI are white crystalline solids and Compounds I, II, IV and VII are oils. No attempt was made to purify the oils, inasmuch as they could be satisfactorily nitrated to give the desired postnitrated polynitrocarbamates. The results are summarized in Table I.

*Collucci, Can. J. Research 23B, 111 (1945).

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II Technical Progress, B (cont.)

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TABLE I

ALIPHATIC POLYNITROCARBAMATES

Isocyanate	Alcohol	Polynitrocarbamate	Yield %	M.P. °C	Recrystallization Solvent	Formula	Analyses		
							Calculated	Found	
NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{NCO}$ NO_2	$\text{HOCH}_2(\text{CH}_2)_3\text{NO}_2$	NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{NCO}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{NO}_2$ NO_2	Quant.	Oil	—	$\text{C}_9\text{H}_{13}\text{N}_5\text{O}_{10}$	—	—	—
NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{NCO}$ NO_2	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$	NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{NCO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ NO_2	Quant.	Oil	—	$\text{C}_7\text{H}_{10}\text{N}_6\text{O}_{12}$	—	—	—
$(\text{NO}_2)_3\text{CH}_2\text{CH}_2\text{NCO}$	$\text{HOCH}_2\text{CH}_2\text{NO}_2$	$(\text{NO}_2)_3\text{CH}_2\text{CH}_2\text{NCO}_2\text{CH}_2\text{CH}_2\text{NO}_2$	91.7	103-104	Chloroform	$\text{C}_7\text{H}_{10}\text{N}_6\text{O}_{12}$	23.31	23.33	22.98 2.80 22.90
$(\text{NO}_2)_3\text{CH}_2\text{CH}_2\text{NCO}$	NO_2 $\text{C}(\text{CH}_2\text{CH}_2)_2\text{NO}_2$	$(\text{NO}_2)_3\text{CH}_2\text{CH}_2\text{NCO}_2\text{CH}_2\text{C}(\text{CH}_2\text{CH}_2)_2\text{NO}_2$	Quant.	Oil	—	$\text{C}_{11}\text{H}_{14}\text{N}_6\text{O}_{20}$	—	—	—
NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{NCO}$ NO_2	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$	NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{NCO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ NO_2	92.5	130-131	Styrene dichloride	$\text{C}_{10}\text{H}_{14}\text{N}_6\text{O}_{18}$	21.36	21.53	21.91 2.70 25.38
NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{NCO}$ NO_2	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$	NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{NCO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ NO_2	91.3	157-159	Methanol-water	$\text{C}_{11}\text{H}_{16}\text{N}_6\text{O}_{16}$	21.06	21.27	21.34 2.59 25.37
CH_3NCO	$\text{HOCH}_2\text{C}(\text{NO}_2)_3$	$\text{CH}_3\text{NCO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$	Quant.	Oil	—	$\text{C}_4\text{H}_6\text{N}_4\text{O}_8$	—	—	—

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II Technical Progress (cont.)

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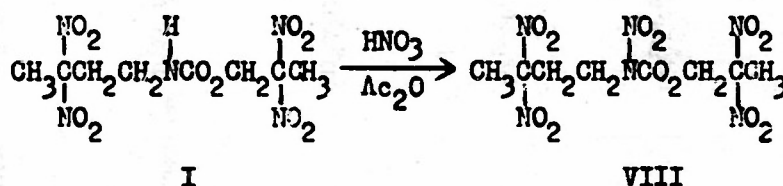
C. PREPARATION OF POSTNITRATED ALIPHATIC POLYNITROCARBAMATES

1. Discussion

a. The following postnitrated aliphatic polynitrocarbammates were prepared by the nitration of the nitrocarbammates described in Section I, B with a mixture of 100% nitric acid and acetic anhydride at 5 to 10°C.

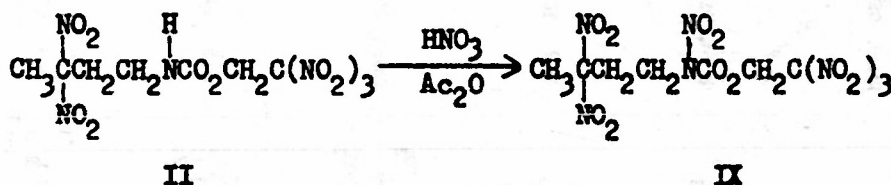
(1) 2,2,6,9,9-Pentanitro-4-oxa-5-keto-6-aza-decane

(VIII):

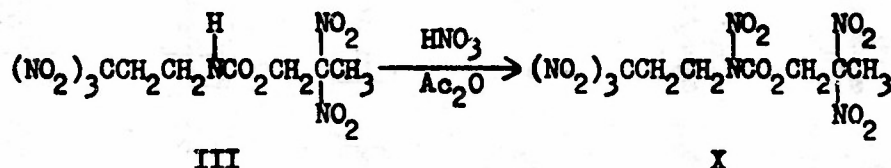


(2) 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-

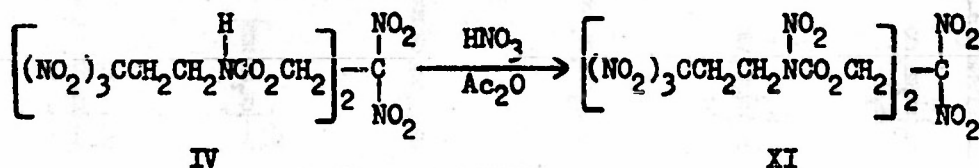
5-aza-nonane (IX):



(3) 2,2,6,9,9,9-Hexanitro-4-oxa-5-keto-6-aza-nonane (X):



(4) 1,1,1,4,8,8,12,15,15,15-Decanitro-4,12-diaza-5,11-diketo-6,10-dioxa-pentadecane (XI):

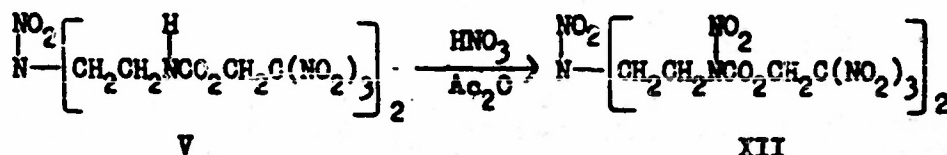


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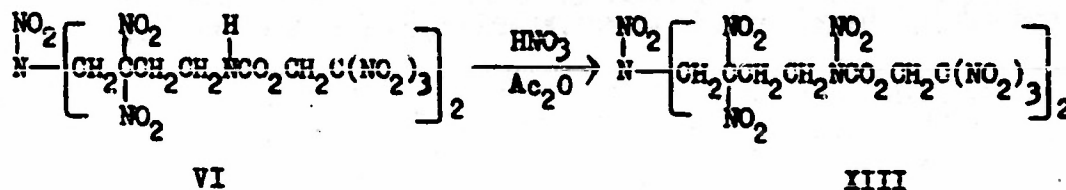
II Technical Progress, C (cont.)

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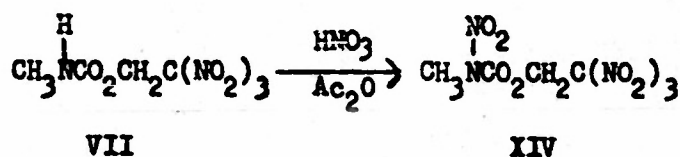
(5) 1,1,1,5,8,11,15,15-Nonanitro-3,13-dioxo-4,12-diketo-5,8,11-triaza-pentadecane (XII):



(6) 1,1,1,5,8,8,10,12,12,15,19,19,19-Tridecanitro-3,17-dioxo-4,16-diketo-5,10,15-triaza-nonadecane (XIII):



(7) 1,1,1,5-Tetranitro-3-oxo-4-keto-5-azahexane (XIV):



2. Experimental

a. The general procedure used was to cool the 100% nitric acid to 0 to 5°C and add the acetic anhydride dropwise, keeping the temperature below 10°C. The solid nitrocarbamate was added portionwise at 5 to 10°C. (Ten ml of 100% nitric acid and 10 ml of acetic anhydride was used per gram of nitrocarbamate.) The solid dissolved readily, and the solution was stirred for 20 min at 5 to 10°C and poured on ice. The white solid was collected, washed with water, and dried in vacuo over potassium hydroxide.

b. In the instances in which the nitrocarbamate was an oil (Compounds I, II, IV, and VII), the oil was dissolved in the acetic anhydride and the solution was added dropwise to the 100% nitric acid, keeping the temperature at 5 to 10°C.

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II Technical Progress, C (cont.)

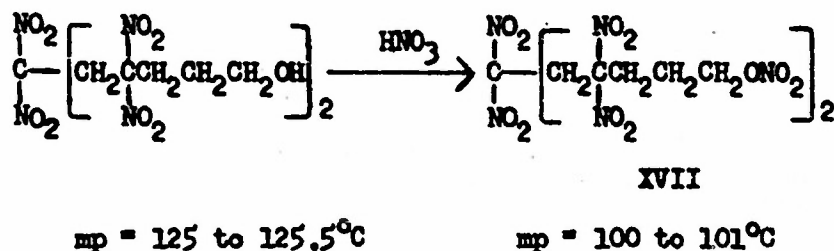
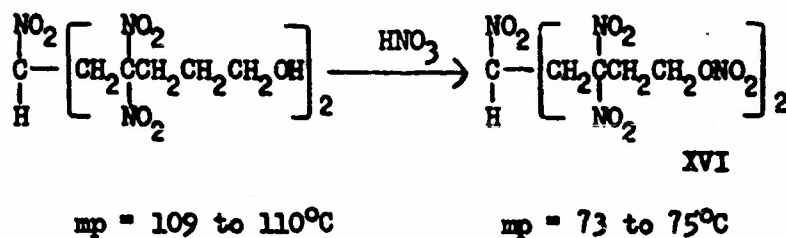
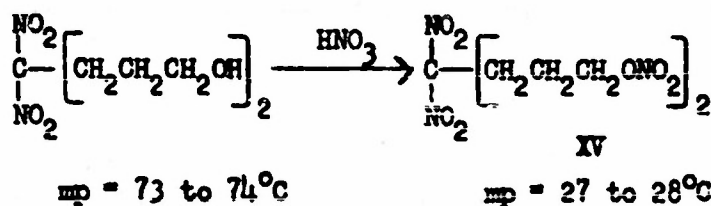
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c. The experimental results are summarized in Table II.

D. PREPARATION OF ALIPHATIC POLYNITRONITRATES

1. Discussion

a. A number of polynitrodiols have been synthesized on the nitropolymer program as intermediates for the preparation of condensation polymers.* It was of interest to the explosives program to convert these polynitrodiols to the corresponding polynitrodinitrates and evaluate these compounds as high explosives. Accordingly, the following nitrates were prepared: 4,4-dinitro-1,7-heptane-dinitrate (XV), 4,4,6,8,8-pentanitro-1,11-undecanedinitrate (XVI), 4,4,6,6,8,8-hexanitro-1,11-undecanedinitrate (XVII), and 4,4,6,8,8-pentanitro-6-aza-1,11-undecanedinitrate (XVIII).



*Aerojet Reports No. 563, 638.

TABLE II

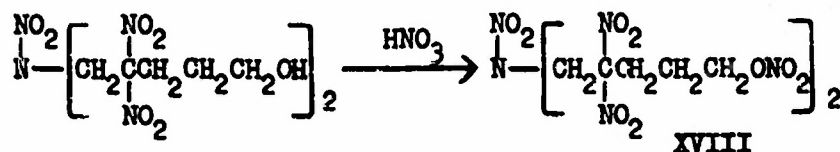
POSTNITRATED ALIPHATIC POLYNITROCARBAMATES

Postnitrated Polynitrocarbamate	Yield %	MP, °C	Recrystallization Solvent	Formula	Analyses				Heat of Combustion cal/g	Calculated Lead-Block Value (PDX = 157)	Calculated Ballistic Mortar Value (RDX = 150)			
					Calculated	Found	Found	Found						
C	H	N	O											
$\text{CH}_3\text{C}(\text{NO}_2)_2\text{CH}_2\text{CH}_2\text{NO}_2$	92.8	70-72	Chloroform	$\text{C}_8\text{H}_{12}\text{N}_6\text{O}_{12}$	25.01	3.15	21.88	25.96	3.36	21.73	2666	2643	117	126
$\text{CH}_3\text{C}(\text{NO}_2)_2\text{CH}_2\text{CH}_2\text{CH}_2\text{NO}_2$	110.5	107-108	Chloroform	$\text{C}_7\text{H}_9\text{N}_7\text{O}_{11}$	20.25	2.19	23.62	20.26	2.21	23.67	2032	2007	153	140
$(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{NO}_2$	83.7	71-72	Chloroform	$\text{C}_7\text{H}_9\text{N}_7\text{O}_{11}$	20.25	2.19	23.62	20.51	2.22	24.05	2033	2051	153	140
$(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{CH}_2\text{NO}_2$	89.7	94-96	Chloroform	$\text{C}_{11}\text{H}_{12}\text{N}_6\text{O}_{24}$	18.97	1.74	24.14	19.29	2.00	24.21	1784	1834	164	132
$(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{NO}_2$	86.3	120-122	Ethylene dichloride	$\text{C}_{10}\text{H}_{12}\text{N}_6\text{O}_{22}$	18.41	1.86	25.77	18.98	2.23	25.87	1792	1826	162	140
$(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{NO}_2$	93.3	150-151	Ethylene dichloride	$\text{C}_{11}\text{H}_{16}\text{N}_6\text{O}_{30}$	18.93	1.82	25.23	19.12	1.84	25.19	1857	1958 1946	164	142
$(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{NO}_2$	74.3	39-40	Diisopropyl ether	$\text{C}_{11}\text{H}_{15}\text{N}_6\text{O}_{30}$	16.97	1.78	24.74	17.29	1.86	24.91	—	—	168	137

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mp = 106 to 108°C

mp = 133 to 135°C

b. Generally nitrates have a lower melting point than the corresponding alcohols. This is confirmed by the behavior of Compounds XV, XVI, and XVII, which melt 25 to 36 degrees lower than the diols from which they were prepared. However, Compound XVIII melts 27 degrees higher than the corresponding diol, a significant factor which must be attributed to the presence of the nitramino group. This is of interest because it is believed that there is a correlation between the melting point and thermal stability of an explosive, the higher-melting compound being more stable provided that other factors are the same.

c. Nitration of 3,3-dinitro-1,5-pentanediol (mp 71 to 72°C), 2,2,4,4,-tetranitro-1,5-pentanediol (mp 97 to 99°C), and 2,2-dinitro-1,3-propanediol (mp ca. 140°C) gave oils which were not investigated further.

2. Experimental

The general procedure was to add the solid polynitrodiol by portions to a tenfold excess of 100% commercial nitric acid at ice-bath temperature. The acid solution was stirred for 15 min at 5 to 10°C and poured on ice. The white solid was collected, washed with water, and dried in vacuo over potassium hydroxide. The results are summarized in Table III.

TABLE III

ALIPHATIC POLYNITRONITRATES

Compound	Yield %	mp, °C	Recrystallisation Solvent	Formula	Analyses			Heat of Combustion cal/g		Calculated Lead-Block Value (RDX = 157)	Calculated Ballistic Mortar Value (RDX = 150)			
					Calculated			Found						
					C	H	N	C	H			N	Calc'd	Found
$\text{NO}_2 \left[\begin{array}{c} \text{NO}_2 \\ \\ \text{C} - \text{CH}_2\text{CH}_2\text{CH}_2\text{ONO}_2 \\ \\ \text{NO}_2 \end{array} \right]_2$	94.7	27-28	Methanol	$\text{C}_7\text{H}_{12}\text{N}_4\text{O}_{10}$	26.93	3.88	17.95	27.17	4.15	18.40	3103	3015	120	132
$\text{NO}_2 \left[\begin{array}{c} \text{NO}_2 \\ \\ \text{C} - \text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{ONO}_2 \\ \\ \text{NO}_2 \end{array} \right]_2$	95.1	73-75	Methanol	$\text{C}_{11}\text{H}_{16}\text{N}_6\text{O}_{16}$	26.25	3.41	19.48	26.79	3.64	19.18	—	—	126	137
$\text{NO}_2 \left[\begin{array}{c} \text{NO}_2 \\ \\ \text{C} - \text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{ONO}_2 \\ \\ \text{NO}_2 \end{array} \right]_2$	95.2	100-102	Methanol	$\text{C}_{11}\text{H}_{16}\text{N}_6\text{O}_{16}$	24.09	2.94	20.44	24.54	2.99	19.71	2643	2600	138	143
$\text{NO}_2 \left[\begin{array}{c} \text{NO}_2 \\ \\ \text{C} - \text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{ONO}_2 \\ \\ \text{NO}_2 \end{array} \right]_2$	95.0	133-135	Methanol	$\text{C}_{11}\text{H}_{16}\text{N}_6\text{O}_{16}$	23.82	3.20	22.22	23.88	2.96	22.15	2727	2726	135	142

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II Technical Progress (cont.)

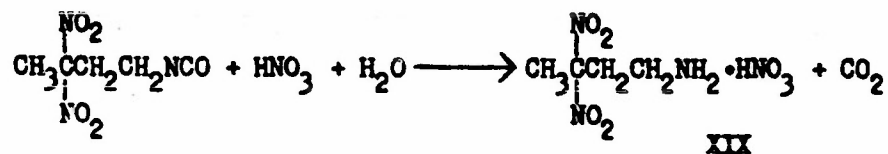
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E. PREPARATION OF ALIPHATIC POLYNITROAMINE SALTS

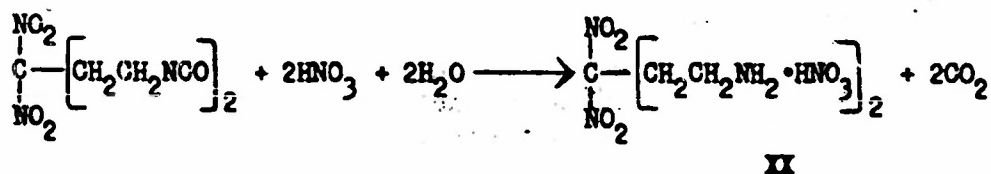
1. Discussion

a. Treatment of 3,3,3-trinitropropyl isocyanate with 35% nitric acid readily gave a quantitative yield of 3,3,3-trinitropropyl ammonium nitrate.* This nitrate salt was of interest for evaluation as a potential high explosive and also as an intermediate for the production of Mannich condensation products. This work suggested the conversion of the isocyanates prepared on the nitropolymer program to the corresponding nitrate salts. Accordingly, the following nitrate salts were prepared:

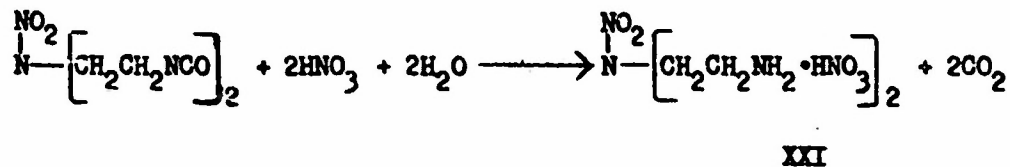
(1) 3,3-Dinitrobutyl Ammonium Nitrate (XIX):



(2) 3,3-Dinitro-1,5-Pentane Diammonium Dinitrate (XX):



(3) 3-Nitroso-1,5-Pentane Diammonium Dinitrate (XXI):



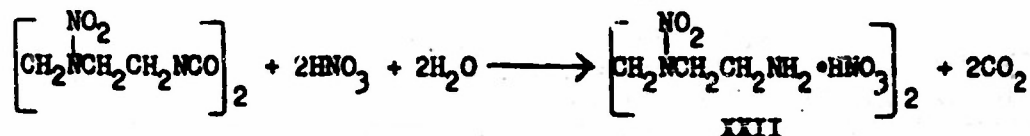
*Aerojet Report No. 682, p. 9.

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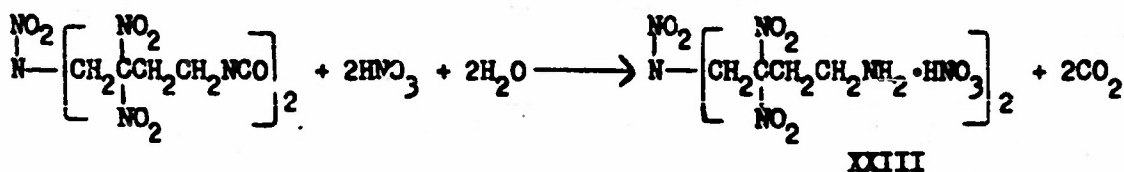
II Technical Progress, E (cont.)

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(4) 3,6-Dinitro-1,8-Octane Diammonium Dinitrate (XXII):



(5) 3,3,5,7,7-Pentanitro-5-aza-1,9-nonane Diammonium Dinitrate (XXIII):



The 3,3,5,7,7-pentanitro-5-aza-1,9-nonane diammonium dihydrochloride (XXIV) was also prepared.

2. Experimental

The general procedure was to heat the isocyanate with a tenfold to fifteenfold excess of 35% nitric acid or concentrated hydrochloric acid on the steam bath for 3 hr. When the solution was cooled, or concentrated if necessary, a quantitative yield of the polynitroamine salt separated. The results are summarized in Table IV.

F. PREPARATION OF SAMPLES FOR EVALUATION BY THE NAVAL ORDNANCE LABORATORY

1. The following samples were prepared and submitted to the Naval Ordnance Laboratory for evaluation:

- a. 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane
- b. 1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-5,7-diaza-decane
- c. 4,4,6,8,8-Pentanitro-1,11-undecane-dinitrate
- d. 4,4,6,6,8,8-Hexanitro-1,11-undecane-dinitrate
- e. 4,4,6,8,8-Pentanitro-6-aza-1,11-undecane-dinitrate
- f. 2,2,6,9,9-Pentanitro-4-oxa-5-keto-6-aza-decane

TABLE IV

ALIPHATIC POLYNITROAMINE SALTS

Compound	mp, °C	Recrystalliz. Solvent	Formula	Analyses					Heat of Combustion cal/g	Calculated Lead-Block Value (RDX=157)	Calculated Ballistic Value (RDX=150)	
				Calculated		Found						
C	H	N	O	Cl	C	H	N	Cl				
NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3$ NO_2	131-133	Methanol	$\text{C}_4\text{H}_{10}\text{N}_6\text{O}_7$	21.24	4.46	24.78	—	21.39	4.63	25.19	—	116
NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3$ NO_2	151-153	Methanol	$\text{C}_5\text{H}_{11}\text{N}_6\text{O}_{10}$	18.87	4.43	26.41	—	19.07	4.70	26.22	—	130
NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3$ NO_2	153-155	Methanol-water	$\text{C}_4\text{H}_{11}\text{N}_6\text{O}_6$	17.52	5.15	30.65	—	17.66	5.11	30.78	—	126
NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3$ NO_2	185-190	Methanol-water	$\text{C}_6\text{H}_{16}\text{N}_6\text{O}_{10}$	19.89	5.01	30.93	—	20.56	5.01	30.78	—	125
NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3$ NO_2	175-180	Concentrated nitric acid	$\text{C}_8\text{H}_{18}\text{N}_6\text{O}_{16}$	18.83	3.56	27.45	—	18.95	3.88	27.10	—	144
NO_2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2 \cdot \text{HNO}_3$ NO_2	173-178	Methanol-water	$\text{C}_8\text{H}_{18}\text{N}_6\text{O}_{10}\text{Cl}_2$	21.02	3.97	24.51	15.51	20.51	4.47	25.40	14.97	—

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II Technical Progress, F (cont.)

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- g. 2,2,6,9,9,9-Hexanitro-4-oxa-5-keto-6-aza-nonane
- h. 1,1,1,4,8,8,12,15,15,15-Decanitro-4,12-diaza-5,11-diketo-6,10-dioxa-pentadecane
- i. 1,1,1,4,13,16,16,16-Octanitro-4,13-diaza-5,12-diketo-6,11-dioxa-8-hexadecyne
- j. 1,1,1,5,8,11,15,15,15-Nonanitro-3,13-dioxa-4,12-diketo-5,8,11-triaza-pentadecane
- k. 1,1,1,5,8,8,8,10,12,12,15,19,19,19-Tridecanitro-3,17-dioxa-4,16-diketo-5,10,15-triaza-nonadecane
- l. 3,3-Dinitrobutyl ammonium nitrate
- m. 3,3-Dinitro-1,5-pentane diammonium dinitrate
- n. 3-Nitrazo-1,5-pentane diammonium dinitrate
- o. 3,6-Dinitrazo-1,8-octane diammonium dinitrate
- p. 3,3,5,7,7-Pentanitro-5-aza-1,9-nonane diammonium dinitrate

2. The results of the preliminary tests by the Naval Ordnance Laboratory on Compounds A53-11 to A53-21 are summarized in Table V. As with most of the polynitrocarbamates, these compounds have good thermal stability and fair to good density.

3. SPIA data sheets, which are presented in the appendix, have been completed on the following compounds:

- a. 1,3-bis(3',3',3'-Trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine
- b. 1,3,5-tris(3',3',3'-Trinitropropyl)-hexahydro-1,3,5-triazine
- c. 2,2,5,7,7,9,12,12-Octanitro-5,9-diaza-tridecane
- d. 2,2,4,7,7,10,12,12-Octanitro-4,10-diaza-tridecane
- e. N',N'-bis(3,3,3-Trinitropropyl) oxamide
- f. N,N'-Dinitro-N,N'-bis(3,3,3-trinitropropyl) oxamide
- g. 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-5-aza-nonane
- h. 1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-5,7-diaza-decane

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II Technical Progress, F (cont.)

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TABLE V

WOL PRELIMINARY TESTS ON HIGH EXPLOSIVES

Compound	mp. °C	T _d , s mm/2.5 hr	Cryst. Dens. g/cm ³	Ignition Temp °C	Thermal Stability (Gas Vol/g at 100°C)			Calculated Lead-Block Value (BOX = 157)	Calculated Ballistic-Mortar Value (BOX = 150)
					45 hr	72 hr	180 hr		
$\text{CH}_3\text{OCH}_2\text{CH}_2\text{NO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ A53-11	107-108	15 (Class 5)	1.733	221	0.50 (Class I)	—	—	153	140
$\text{CH}_3\text{OCH}_2\text{CH}_2\text{NO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ A53-12	140-141	18 (Class 5)	1.790	212	1.01 (Class I)	—	—	167	144
$\text{H}-\left[\text{CH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{ONO}_2\right]_2$ A53-13	73-75	34 (Class 4)	—	—	—	—	—	126	137
$\text{H}-\left[\text{CH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{ONO}_2\right]_2$ A53-14	100-101	28 (Class 4)	—	—	—	—	—	138	144
$\text{H}-\left[\text{CH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{ONO}_2\right]_2$ A53-15	133-135	85 (Class 2)	—	—	—	—	—	135	142
$\text{CH}_3\text{OCH}_2\text{CH}_2\text{NO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ A53-16	70-72	47 (Class 3)	1.61	293	(Class II)	—	—	117	126
$(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{NO}_2\text{CH}_2\text{C}(\text{NO}_2)_3$ A53-17	66-68	16 (Class 5)	—	229	(Class III)	—	—	153	140
$\text{H}-\left[\text{CH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{C}(\text{NO}_2)_3\right]_2$ A53-18	94-96	20 (Class 5)	1.74	212	5.0 (Class II)	9.2	27.2	164	132
$\left[(\text{NO}_2)_3\text{CCH}_2\text{CH}_2\text{NO}_2\text{CH}_2\text{C}(\text{NO}_2)_3\right]_2$ A53-19	110-111	16 (Class 5)	1.65	192	24 (Class III)	—	—	137	134
$\text{H}-\left[\text{CH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{C}(\text{NO}_2)_3\right]_2$ A53-20	120-122	17 (Class 5)	1.77	220	1.1 (Class I)	1.6	3.5	162	140
$\text{H}-\left[\text{CH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{C}(\text{NO}_2)_3\right]_2$ A53-21	150-151	12 (Class 5)	1.76	217	1.4 (Class I)	1.9	3.3	154	142

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III. DESENSITIZATION OF RDX

A. INVESTIGATION OF COMBINATIONS OF POLYDINITROBUTYL ACRYLATE, PLASTICIZERS, AND SURFACE-ACTIVE AGENTS

1. Discussion

a. In the investigation of new coatings for RDX by the method of preparing composition A, a systematic test of various combinations of a plasticizer and a surface active agent with polydinitrobutyl acrylate is in progress. This study has a dual purpose:

(1) Correlation of the compatibility of such combinations with their desensitizing effect.

(2) Pointing out new, promising combinations for further consideration.

For the plasticizers, four commercially used compounds, along with 2,2-dinitropropane, were chosen. The surface-active agents were seven ester-type materials selected as the best of the many agents tested in the past. Each combination was made up and tested for compatibility. As the next step, specimens of RDX coated with the same combinations are being prepared, and their impact stabilities determined.

b. Every attempt was made to insure uniform, reproducible conditions. Fixed proportions were used throughout. The polymer had been worked up by reprecipitating, to eliminate light fractions that can themselves provide a plasticizing action.

c. Present results are too sketchy for correlation of compatibility with desensitizing action. With respect to the second purpose, these results have already confirmed the softening action of several plasticizers in improving the efficacy of polydinitrobutyl acrylate coating. The results have also confirmed earlier observations* that some alternate surface-active agents are as good as or better than Span 85.

d. Some further experiments were performed with the aim of increasing the softness of the coating and at the same time increasing its oxygen balance (thus increasing explosive strength). Using only Atlas G-3785 surface-active agent, these experiments employed the copolymer of dinitrobutyl acrylate and mono nitrobutyl acrylate for increased softness, and also varied the amounts of the constituents. These experiments show the possibility of appreciably raising the oxygen balance with increased amounts of 2,2-dinitropropane. They also indicate that 1% of G-3785 surface-active agent performs as well as 2.5%, and has the advantage of higher oxygen balance. However, none of the coating formulations gave as good desensitization as is attained with Composition A.

*Aerojet Report No. 682, et seq.

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III Desensitisation of RDX, A (cont.)

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2. Experimental

a. Specimens composed of polydinitrobutyl acrylate, plasticizer, and surface-active agent were made up in the ratio 8:2:3.3. This ratio gives a content of surface-active agent slightly in excess of the amount ordinarily used in coating; the actual coating compositions are thus bracketed between specimens containing surface-active agents and those containing none. These specimens were dissolved in acetone and the solutions were dropped on a glass plate. The solvent was evaporated and the residual films were inspected and compared. The three qualities selected for rating the films were opacity, separation, and fragility. The results are given in Table VI.

b. A parallel experiment was begun, in which RDX is coated with the same combinations of polydinitrobutyl acrylate, plasticizer, and surface-active agent in the ratio 8:2:2.5% RDX. The procedure was the previously described adaptation of the process for making Composition A,* except that the amount of RDX used was reduced to 4.0 g, and the other amounts were reduced proportionately. The products were evaluated by measuring impact stabilities on the Bureau of Mines apparatus with the 2-kg weight, using the bare anvil and also (sometimes) 5/0 sandpaper. Results of these tests are shown in Table VII.

c. Some additional coating tests were performed using similar materials but falling outside the experimental pattern described above. Results of these tests are given in Table VIII. Oxygen balances of these formulations were calculated, and are also tabulated here.

d. All polymers used had been thoroughly cured, and then worked up by reprecipitating twice from acetone solution in methanol. This treatment removed fractions of low molecular weight.

*Aerojet Report No. 682, p. 21.

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III Desensitization of EDX, A (cont.)

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TABLE VI

COMPATIBILITIES OF SYSTEMS OF POLYDINITROBUTYL
ACRYLATE, PLASTICIZER, AND SURFACE-ACTIVE AGENT

Films contain 8 parts PDNBA
 2 parts plasticizer
 3.3 parts surface-active agent

Note: All samples were inspected for opacity (first column of numerals); separation (second column); and fragility (third column), and rated by a 1-2-3 system, where 1 denotes least, 2 medium, and 3 most.

Plasti- cizer	Surface-Active Agent							
	--	Span 85	Glyc. Mono 0	Prosol 307	G-949	G-1077	G-2135	G-3785
--	1,1,3	2,3,3	3,2,3	3,1,3	2,3,3	3,2,3	3,2,3	3,1,3
DOS	3,1,3	3,3,3	3,3,3	3,3,3	2,3,3	3,2,3	3,3,3	3,1,3
DBS	3,1,1	3,3,3	3,2,3	3,2,3	2,3,3	3,2,3	3,2,3	3,2,1
DOP	2,1,2	2,3,3	3,2,3	3,2,3	2,3,3	3,2,3	3,2,3	3,2,3
TCP	1,1,1	2,3,2	1,1,3	1,2,3	1,3,2	2,2,3	3,2,2	3,1,2
DNP	1,1,2	2,3,2	3,2,2	3,1,2	1,3,3	3,2,2	3,2,2	3,1,3

DOS is dioctyl sebacate

DBS is dibutyl sebacate

DOP is dioctyl phthalate

TCP is tricresyl phosphate

DNP is 2,2-dinitropropane

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III Desensitization of RDX, A (cont.)

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TABLE VII

IMPACT STABILITIES OF SYSTEMS COMPOSED OF POLYDINITROBUTYL ACRYLATE, PLASTICIZER, AND SURFACE-ACTIVE AGENT

Results in parentheses determined on 5/0
sandpaper; others on bare anvil.

Impact Stability of Systems Containing The Following Surface-Active Agents, cm/2 kg								
Plasti- cizer	—	Span 85	Glyc. Mono O	Prosol 307	G-949	G-1077	G-2139	G-3785
—	35-40	85	—	>100 to 45-50*	—	—	—	90-100
DOS	—	—	—	—	—	—	—	—
DBS	60-75	>100	—	—	—	—	—	>100 (15-20)
DOP	—	—	—	—	—	—	—	—
TCP	80-85	75-80	<55	—	—	—	—	>95
DNP	50-55	>100	—	55-60	—	75-80	—	>95, >100

Polydinitrobutyl acrylate, plasticizer, and surface active agent in the
ratio 8:2:2.5% RDX.

Reference: RDX = 40 (10-15)

Comp. A-3, > 100 (45-50)

*Large variations among separate preparations.

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III Desensitization of RDX, A (cont.)

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TABLE VIII

MISCELLANEOUS DESENSITIZATION EXPERIMENTS

Run	Coating	Oxygen* Balance	I.S., cm/2 kg	
			Bare	On paper
6/18/1	5% PDNBA/5% DNP/1% G-3785	-29	>100	10-15
6/18/2	5% PDNBA/5% DNP/2.5% G-3785	-32	>100	10-15
6/18/3	7.5% PDNBA/7.5% DNP/2.5% G-3785	-34	>100	10-15
6/5/1	8% Copolymer/2% DBS/2.5% G-3785	-37	>100	20-25
6/19/1	6% Copolymer/2% DBS/2% DNP/G-3785	-34	>100	25
RDX		-22	40	10-15
Comp. A-3		-43	>100	40-45

Copolymer made from 50% dinitrobutyl acrylate and 50% mono-nitrobutyl acrylate.

*Oxygen balances calculated by assuming a reasonable average formula for all surface-active agents.

CONFIDENTIAL

III Desensitization of RDX (cont.)

Report No. 737

B. SURFACE-ACTIVE AGENTS CONTAINING NITRO GROUPS

1. Discussion

a. Previous trials of many surface-active agents for use in coating RDX have indicated that certain agents are most efficacious.* These had the common feature of being esters of polyols and oleic acid, or other long-chain fatty acids. In fact, such agents by themselves can provide good temporary desensitization (temporary because of the drainage of liquid coatings). These results suggested the use of similar compounds containing substituted nitro groups, either as surface-active agents or as coatings per se.

b. The first compound whose synthesis was attempted was the monocarbamate of 3,3,3-trinitropropyl isocyanate and glycerol mono oleate; this product was a viscous liquid which could not be crystallized. This circumstance, along with the mixed nature of the original oleate, might well lead to a mixed product, and analysis confirmed that the product was considerably different from the simple monocarbamate. The product was subjected to the adsorption test described in Report No. 682, in which the amount adsorbed on RDX from an acetone-water system is measured under standardized conditions. Next, a coating experiment was performed, using the method of preparation of Composition A.** Application of 10% of the product alone gave a coated specimen whose impact stability was approximately as good as that of Composition A. When used as a surface active agent, in conjunction with polydinitrobutyl acrylate coating, the product was comparable to the agents now in use.

c. The preparation of chemically similar solid compounds will be attempted next.

2. Experimental

a. The reaction between glycerol mono oleate and 3,3,3-trinitropropyl isocyanate was carried out in the same manner as described in II,B,2. The product was a viscous liquid:

Anal. Calc'd for monocarbamate $C_{25}H_{43}N_4O_{11}$: %C, 52.16; %H, 7.53; %N, 9.74

Found: %C, 70.44; %H, 7.54; %N, 8.55

*Aerojet Reports No. 589, 682.

**Aerojet Report No. 682, p. 21.

CONFIDENTIAL

III Desensitization of RDX, B (cont.)

Report No. 737

b. The product was subjected to the standard adsorption test for surface-active agent. Results, compared with the values for Span 85, which is known to be a good surface-active agent, are shown below:

Adsorption of 0.15 g Agent on 3.00 g RDX from a Medium
of 5 ml Acetone and 10 ml Water

	<u>Appearance of Agent in Medium</u>	<u>% Adsorbed</u>	<u>Impact Stab.,* cm/2 kg</u>
Carbamate	Suspension and oil layer	81	65-75
Span 85	Suspension	52-62	>100**

*For carbamate alone, >100; for RDX, 30.

**Value for RDX from a different lot.

c. The new material may be too oily, from its behavior in the test; the coating on the RDX was noticed to be somewhat uneven. This degree of desensitization is fair though not as good, per unit amount adsorbed, as that obtained with the best surface-active agents described in Report No. 682.

d. Using the standard coating procedure, 10% of this product was applied to RDX. In another experiment it was used in conjunction with polydinitrobutyl acrylate, replacing both the plasticiser and the surface-active agent used in the systematic experiments described in the preceding section. Impact stabilities of the products were measured; the results are:

<u>Coating</u>	<u>Impact Stability, cm/2 kg</u>	
	<u>Bare</u>	<u>On Paper</u>
10% Carbamate	>100	40
8% PDNBA/4.5% carbamate	>100	20-25
— RDX	40	10-15
— Comp. A-3	>100	40-45

e. The RDX used in these experiments was Wabash L-119.

CONFIDENTIAL

Data Questionnaire on

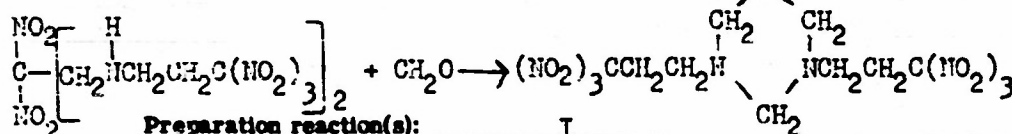
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, API/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,3-bis(3',3',3'-Trinitropropyl)-

Name 5,5-dinitro-hexahydro-1,3-diazineEmpirical formula (C₁₀H₁₂N₁₀O₁₆)Structure (configuration) I

Information submitted by:

Activity Aerojet-General CorporationPerson M. B. Frankel and L. T. CarletonDate 1 August 1953Preparation reaction(s): I

1. Quantitative analysis:(% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	22.65	2.66		26.42		
By determination	22.95	2.65		26.72		

2. Burning properties:(compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL OSRD 3401 p.8	2.5-kg weight
b. Thermal Stability	OSRD 3401 p.8	
c. Vacuum Stability	NOL OSRD 3401 p.8	7 hr at 80°C
d. Temperature of Explosion	OSRD 3401 p.8	
e. Temperature of Ignition	NOL OSRD 3401 p.8	
f. Impact Stability	Bureau of Mines Bull. No. 346, p. 72 (2-kg weight)	
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetryl, H.C., etc.)

New Compound test results

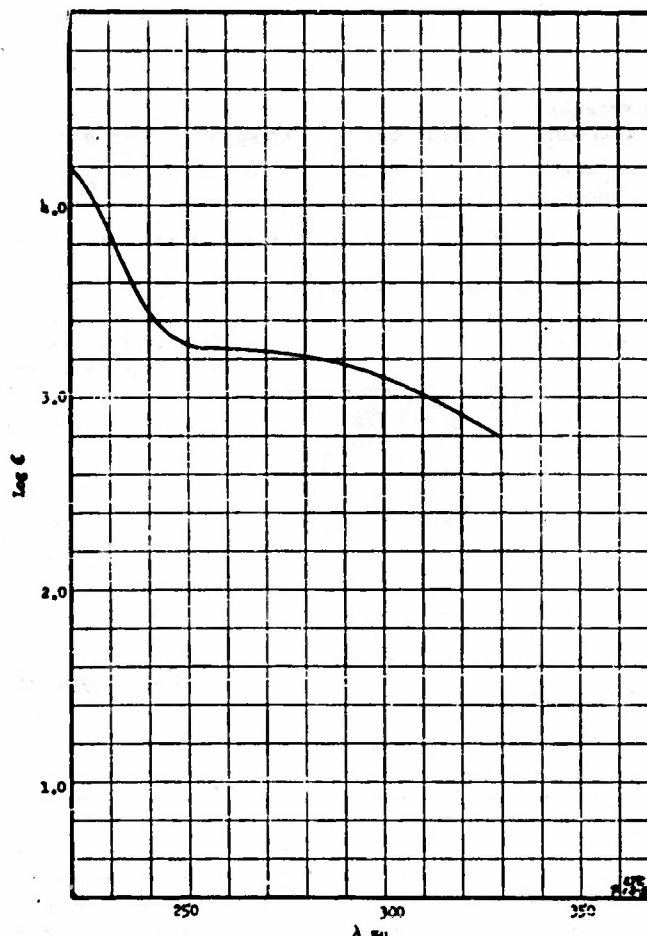
a. Tetryl, 32 cm/2.5 kg	20 cm/2.5 kg
b.	
c.	>30 cc/g
d.	
e.	129°C
f. BDY, 30 cm/2 kg	25-30 cm/2 kg
g.	
h.	

4. Heat of formation: (ΔH) \pm -20 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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Report No. 737
SPIA/M3

- | | By Experiment | By Calculation | Method
Description or reference. Separate sheet if necessary. |
|--|--|--|--|
| 5. Energy of explosion (Q)
(at 250°C. H ₂ O liquid) | | cal/gm | |
| 6. Heat of combustion (H _c)
(at 250°C. H ₂ O liquid) | 2637 | cal/gm | 2637 |
| 7. Specific impulse (I _{sp}) calc: | | lb-sec/lb | |
| 8. Physical form of compound (viscous liquid, crystalline type, etc.) | needle-shaped crystals | | |
| 9. Simple microscope analysis data:
(crystal studies) | | | |
| 10. Density (micro method) | 1.683 gm/cm ³ | (Micro or other method) | gm/cm ³ |
| | (NOL) | (Analyze on separate sheet any unique methods you use.) | |
| 11. Index of refraction: (n _D ^{25°C.}) | | 12. Color | Yellow |
| | | 13. Odor | None |
| 14. pH at 25°C. | 2.1 | (Method reference OSRD 3401 v.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator used or Beckman pH meter.) | |
| | With Beckman pH meter, 0.054 M in acetone/water (5/1 volume ratio) | | |



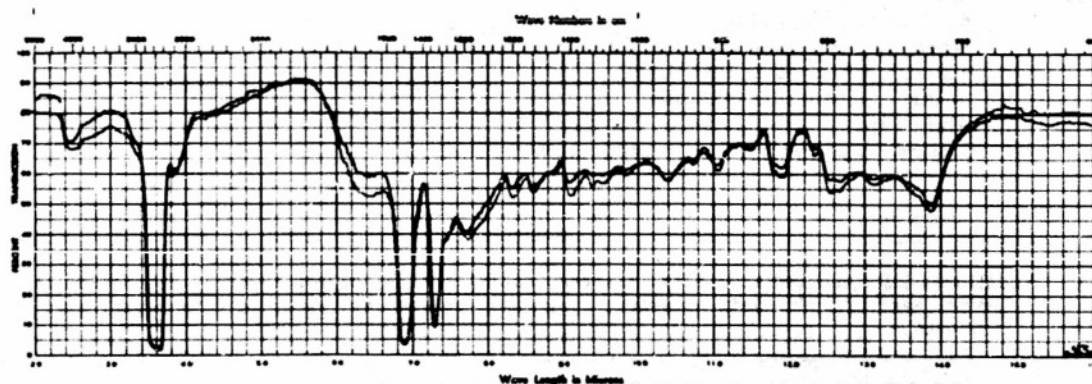
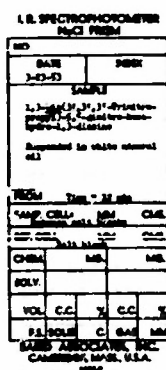
Ultraviolet Absorption Spectrum of 1,3-bis(3',3'-Trinitropropyl)-5,5-dinitro-hexahydro-1,3-diazine in Ethanol

20. Melting point: 119-121 °C. (d)

TEMPERATURE °C.

TIME

<u>0.1</u>	g/100 ml H ₂ O at 25°C.	<u> </u>	g/100 ml H ₂ O at <u> </u> °C.
<u>160</u>	g/100 ml <u>acetone</u>	at <u>25</u>	°C.
	(name material used as solvent)		
<u>4.3</u>	g/100 ml <u>toluene</u>	at <u>25</u>	°C.
	(name material used as solvent)		



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thickol _____
 Methacrylate _____
 Other compounds _____
30. Availability
a. Amount now available? _____ research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existence, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 153 }
Calculated Ballistic Mortar Value = 157 } Method of Aerojet Report No. 512, p. 8

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Report No. 737
SPIA/MS

Data Questionnaire on

COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound further. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFM/JMU, 8651 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,3,5-tris(3',3',3'-Trinitropropyl)-

Name hexahydro-1,3,5-triazine

Empirical formula C₁₂H₁₂N₆O₁₈

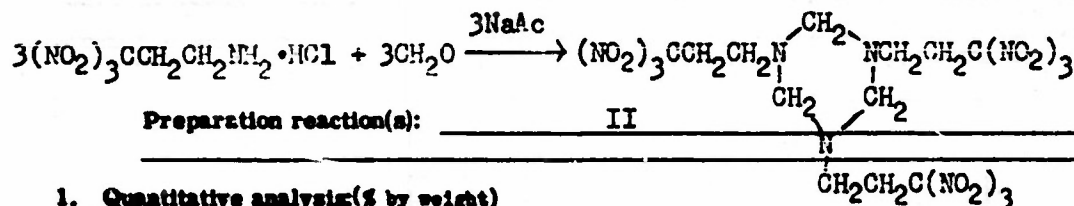
Structure (configuration) II

Information submitted by:

Activity Aerojet-General Corporation

Person M. B. Frankel and L. T. Carleton

Date 1 August 1953



1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen
Calculated from formula	23.31	2.93	27.19	27.19
By determination	23.78	2.68	27.21	27.21

2. Burning properties (compared to nitrocellulose, under nitrogen at atmospheric pressure):

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL XXXXXXXXXX	2.5-kg weight
b. Thermal Stability	OSRD 3401 p.8	
c. Vacuum Stability	NOL XXXXXXXXXX	at 80°C
d. Temperature of Explosion	OSRD 3401 p.8	
e. Temperature of Ignition	NOL XXXXXXXXXX	
f. Impact Stability, Bureau of Mines Bull. No. 346, p. 72 (2-kg weight)		
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetryl, H.C., etc.)

New Compound test results

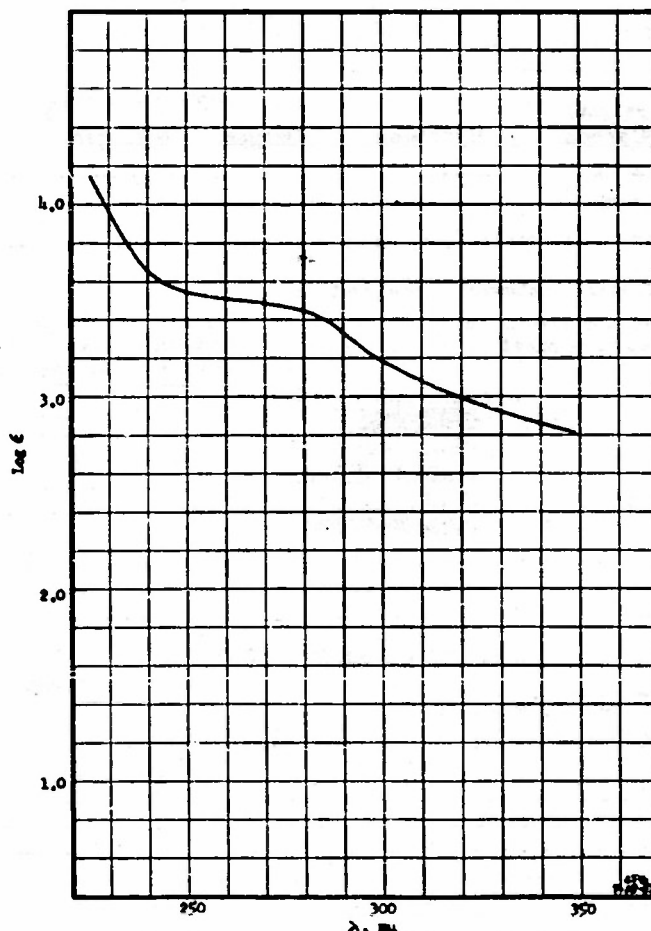
a. Tetryl, 32 cm/2.5 kg	20 cm/2.5 kg
b.	
c.	exploded in 1 hr
d.	
e.	140°C
f. RDX, 30 cm/2 kg	15-20 cm/2 kg
g.	
h.	

4. Heat of formation: (ΔH) \pm -141 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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Report No. 737
SPLA/M3

By Experiment	By Calculation	Method
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)	cal/gm	Description or reference. Separate sheet if necessary.
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2748 cal/gm	2812 Aerojet Report No. 417A
7. Specific impulse (I _{sp}) calc:	lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)		Crystalline Solid
9. Simple microscope analysis data: (crystal studies)		
10. Density (Macro method)	1.657 gm/cm ³ . (Micro or other method)	gm/cm ³ . (NOL) (Include on separate sheet any unique methods you use.)
11. Index of refraction (n _D ^{25°C.})	12. Color	Yellow 13. Odor
		None
14. pH at 25°C. 6.2 (Method reference OESD 3401 v.4, or OESD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator used or Beckman pH meter.)		
pH meter, 0.060 M in acetone/water (5/1 volume ratio) In Beckman		



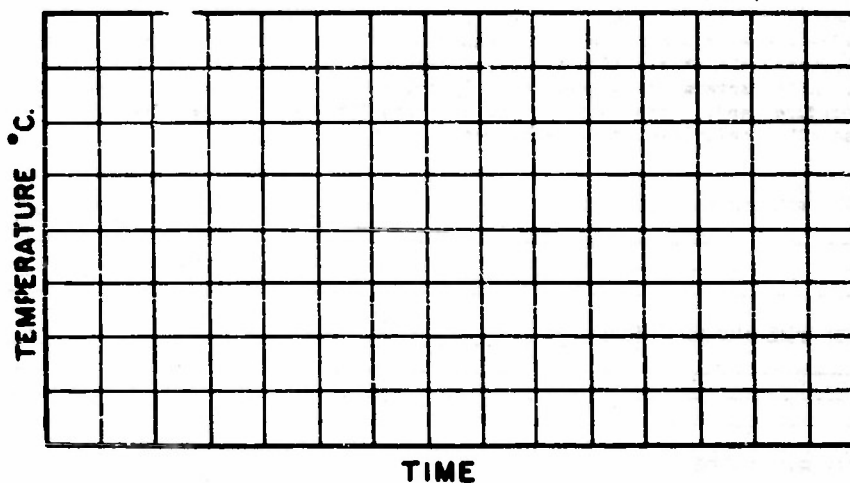
Ultraviolet Absorption Spectrum of 1,3,5-tris(3',3',3'-Trinitropropyl) hexahydro-1,3,5-triazine in Buyl ether

20. Melting point: 120-122 °C.(d)

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Report No. 737
SP1A/M3

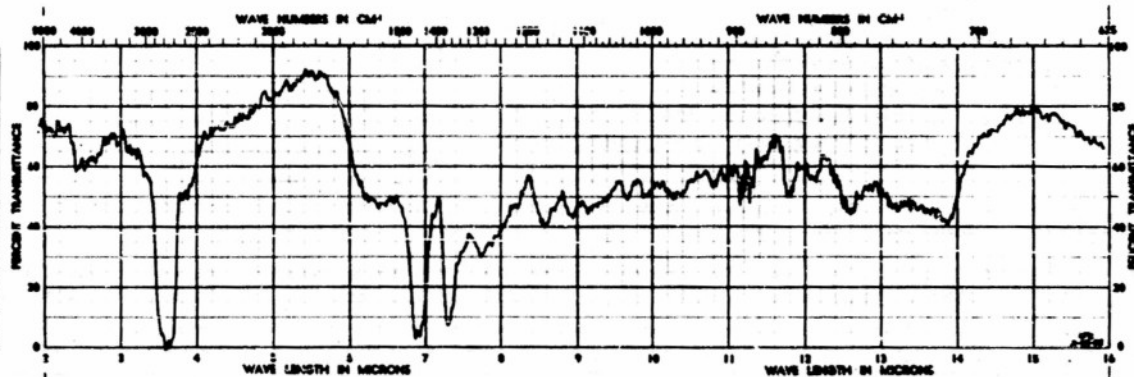
21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
55 g/100 ml acetone at 25 °C.
 (name material used as solvent)
0.2 g/100 ml toluene at 25 °C.
 (name material used as solvent)

I.R. SPECTROPHOTOMETER			
NACI PRISM			
NO.	DATE	SPEC.	
	3-23-53		
SAMPLE			
1,3,5-trisubstituted benzene			
propyl-substituted-1,3,5-			
triazole			
Apparent to white solid			
43			
REMARKS			
Temp. - 25°C.			
SAMP. CH. L.	MM	CHL.	
REF. CHL.	MM	CHL.	
CHINA	MM	MM	
SOLV.			
VOL. CC.	Y	CC.	Y
FA. SOLV.	C	CHL.	MM
BARD ASSOCIATES, INC.			
CAMBRIDGE, MASS., U.S.A.			



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Report No. 737
SP1A/M3

Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSTD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____ : _____

29. Polymerizing properties of the new compound:
 - (a) By itself _____
 - (b) In mixtures (with additives) _____
 - (c) Inhibiting action on polymerization of:
 - Thiokol _____
 - Methacrylate _____
 - Other compounds _____
30. Availability
 - a. Amount now available? research quantities
 - b. When was available material first prepared? _____
 - c. Amount prepared at that time? _____
 - d. Is large production feasible? _____
 - e. Plant capacity in existence, lbs/day? _____
 - f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

 Calculated Lead-Block Value = 130 }
 Calculated Ballistic-Mortar Value = 140 } Method of Aerojet Report No. 512

p. 8

CONFIDENTIAL

Data Questionnaire on

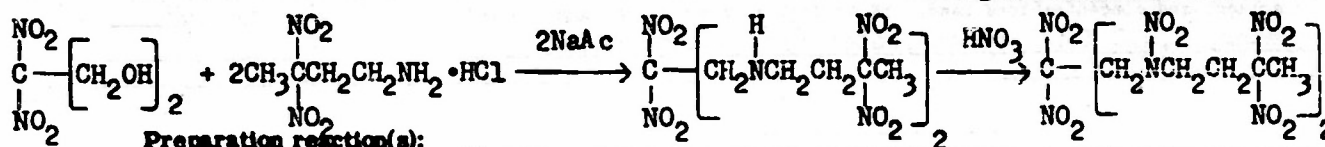
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, APL/JHU, 8631 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 2,2,5,7,7,9,12,12-Octanitro-

Name 5,9-diaza tridecaneEmpirical formula C₁₁H₁₈N₂O₁₆Structure (configuration) III

Information submitted by:

Activity Aerojet-General CorporationPerson H. E. Frankel and L. T. CarletonDate 1 August 1953

Preparation reaction(s):

III

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	24.18	3.32		25.64		
By determination	24.76	3.11		25.52		

2. Burning properties (compared to nitrocellulose, under nitrogen at atmospheric pressure):

(faster? slower? residue? etc.)

3. Stability and Sensitivity. Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL <u>OSRD 3401 p.8</u>	<u>2.5-kg weight</u>
b. Thermal Stability	NOL <u>OSRD 3401 p.8</u>	
c. Vacuum Stability	NOL <u>OSRD 3401 p.8</u>	<u>48 hr at 80°C</u>
d. Temperature of Explosion	NOL <u>OSRD 3401 p.8</u>	
e. Temperature of Ignition	NOL <u>OSRD 3401 p.8</u>	
f. Impact Stability	Bureau of Mines, Bull. No. 346, p. 72 (2-kg weight)	
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetra, H.C., etc.)

New Compound test results

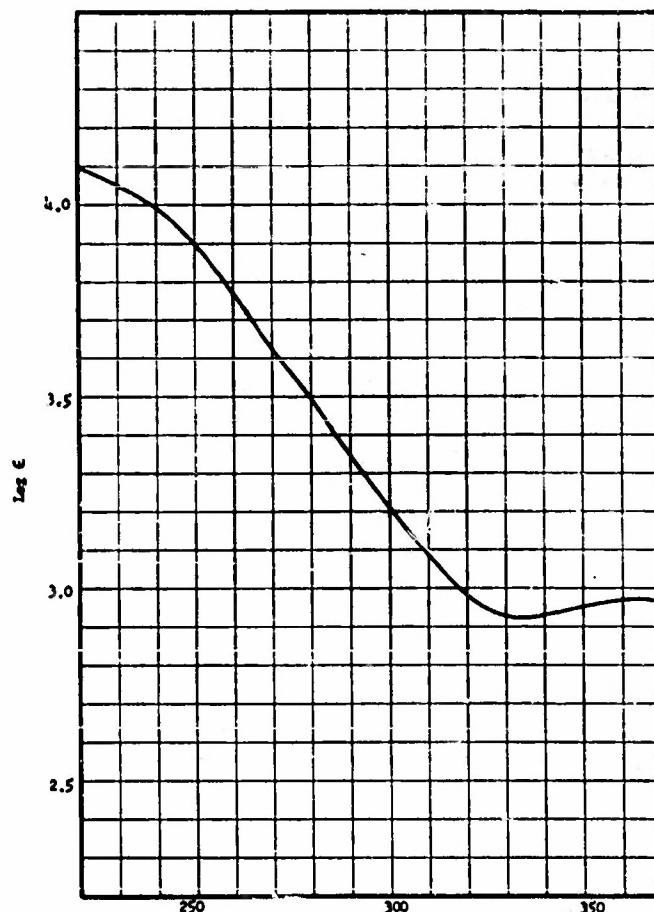
a. Tetra, 32 cm/2.5 kg	37 cm/2.5 kg
b.	
c.	2.2 cc/g
d.	
e.	1800°C
f. RDX, 30 cm/2 kg	10-15 cm/2 kg
g.	
h.	

4. Heat of formation: $(\Delta H) \pm \frac{-62}{(\text{Indicate sign})}$ Kcal. calories at 25°C., 1 atm. pressure

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Report No. 737
SP1A/M3

	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (G) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2897, 2913	cal/gm	2879
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	Fine crystals		
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method)	1.676 gm/cm ³ . (NOL)	(Micro or other method)	gm/cm ³ . (Indicate in separate sheet any unique methods you use.)
11. Index of refraction (n _D ^{25°C.})		12. Color	White
		13. Odor	None
14. pH at 25°C.	5.0	(Method reference OSRD 3401 v.4, or OSRD 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator water or Beckman pH meter.)	
	With Beckman pH meter, 0.030 M in acetone/water (5/1 volume ratio)		



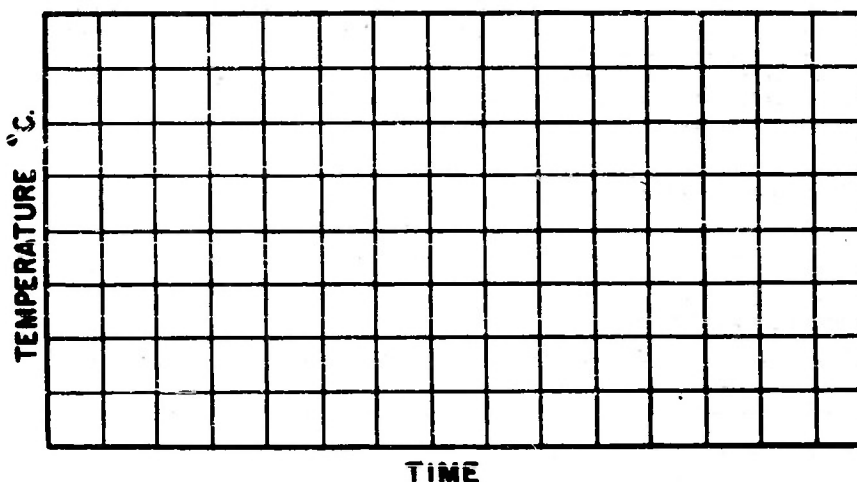
Ultraviolet Absorption Spectrum of 2,2,5,7,7,9,12,12-Octanitro
5,9-dinitro tridecane in Acetonitrile

20. Melting point: 137-143 °C.(d)

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Report No. 737
SFIA/M3

21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)

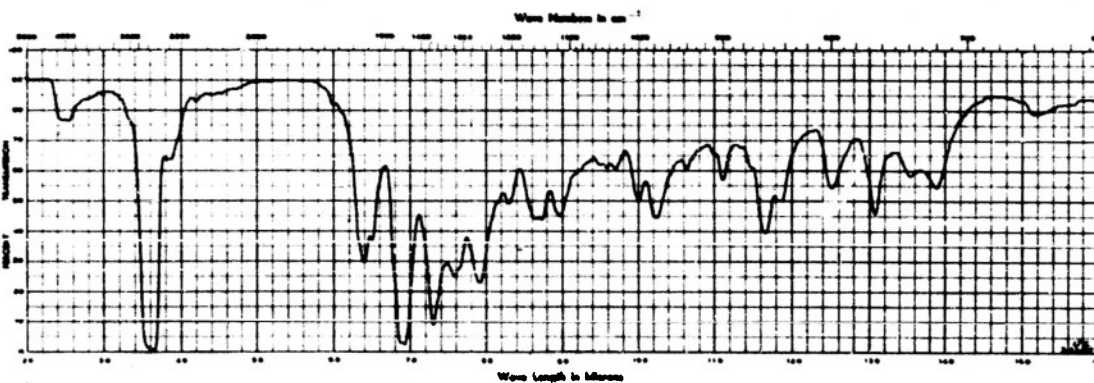


22. Solubility of new compound

0.1 g/100 ml H₂O at 25°C. g/100 ml H₂O at °C.
31 g/100 ml acetone at 25 °C.
 (name material used as solvent)
0.1 g/100 ml toluene at 25 °C.
 (name material used as solvent)

I.R. SPECTROPHOTOMETER
NaCl prism

NO.	DATE	INDEX
1-21-51		
1,2,5,7,9,12,13-dibromotetra- 5,7-diene hydrocarbon Supplied to: R&M Research S.A.		
NAME: <u> </u> Film: <u>12</u> mm		
TEMP. CELL	mm	CHL
REF. CELL	mm	CHL
CHINA	mm	mm
SOLV.		
VOL. CC.	%	CC. %
P.A. SOLV.	C	CHL mm
BARR ASSOCIATES, INC. CAMBRIDGE, MASS., U.S.A.		



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thickol _____
 Methacrylate _____
 Other compounds _____
30. Availability
a. Amount now available? Research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existence, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 128
Calculated Ballistic-Mortar Value = 140 } Method of Aerojet Report No. 512, p. 8

CONFIDENTIAL

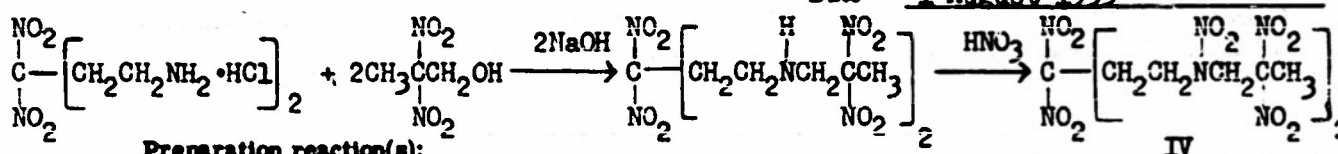
Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFL/JHU, 8631 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 2,2,4,7,7,10,12,12-Octanitro-

Name 4,10-diaza tridecaneEmpirical formula C₁₁H₁₈N₁₀O₁₆Structure (configuration) IV

Information submitted by:

Activity Aerojet-General CorporationPerson M.B. Frankel and L.T. CarletonDate 1 August 1953

Preparation reaction(s):

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	<u>24.18</u>	<u>3.32</u>		<u>25.64</u>		
By determination	<u>24.76</u>	<u>3.35</u>		<u>24.98</u>		

2. Burning properties (compared to nitrocellulose, under nitrogen at atmospheric pressure):

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL <u>OSRD 3401 p.8</u>	<u>2.5-kg weight</u>
b. Thermal Stability		
c. Vacuum Stability	NOL <u>OSRD 3401 p.6</u>	<u>48 hr at 100°C</u>
d. Temperature of Explosion		
e. Temperature of Ignition	NOL <u>OSRD 3401 p.6</u>	
f. Impact Stability	Bureau of Mines Bull. No. <u>346</u> , p. <u>72</u> (2-kg weight)	
g.		
h.		

RESULTS OF ABOVE TESTS

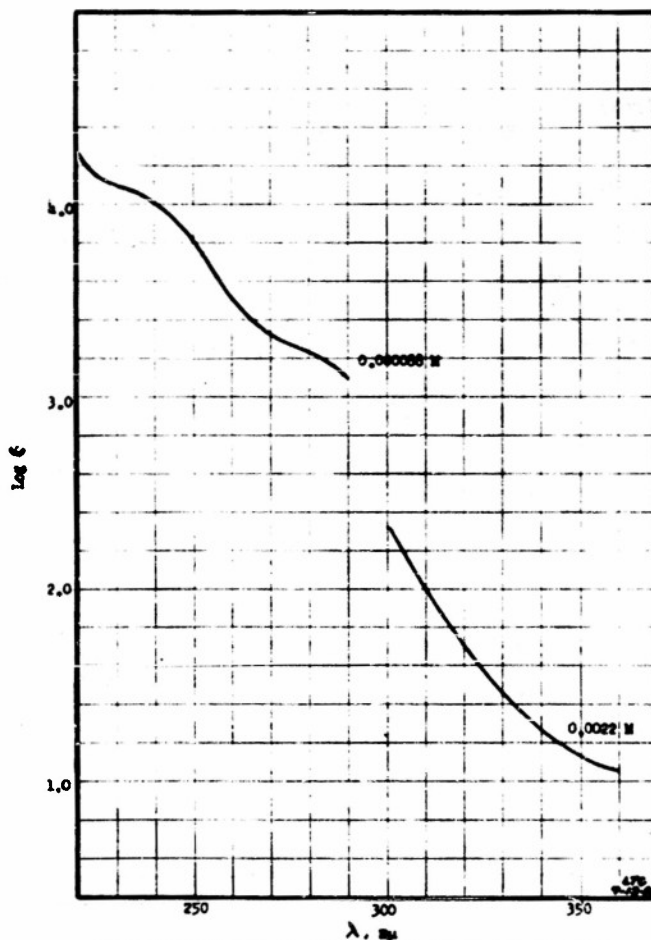
Reference compound (designation-TNT, Tetryl, H.C., etc.)	New Compound test results
a. Tetryl, 32 cm/2.5 kg	<u>44 cm/2.5 kg</u>
b.	
c.	<u>0.7 cc/g</u>
d.	
e.	<u>244°C</u>
f. RDX, 30 cm/2 kg	<u>40-45 cm/2 kg</u>
g.	
h.	

4. Heat of formation: (ΔH_f°) -104 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

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Report No. 737
SPIA/M3

By Experiment	By Calculation	Method <small>Description or reference. Separate sheet if necessary.</small>
5. Energy of explosion (Q) _____ cal/gm _____ <small>(at 2500° F. H₂O liquid)</small>		
6. Heat of combustion (H _c) <u>2830, 2825</u> cal/gm <u>2878</u> <small>(at 2500° F. H₂O liquid)</small>		<u>Aerojet Report No. 417A</u>
7. Specific impulse (I _{sp}) calc: _____ lb-sec/lb _____		
8. Physical form of compound (viscous liquid, crystalline type, etc.) <u>Fine crystals</u>		
9. Simple microscope analysis data: _____ <small>(crystal studies)</small>		
10. Density (Micro method) <u>1.708</u> gm/cm ³ . (Micro or other method) _____ gm/cm ³ . <small>(NOL) (Explain on separate sheet any unique methods you use.)</small>		
11. Index of refraction (n _D ^{25°C.}) _____	12. Color <u>White</u>	13. Odor <u>None</u>
14. pH at 25°C. <u>6.8</u> (Method reference Q88D 3401 v.4, or Q88D 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator meter or Beckman pH meter.) <u>With Beckman pH meter, saturated solution in acetone/water (5/1 volume ratio)</u>		

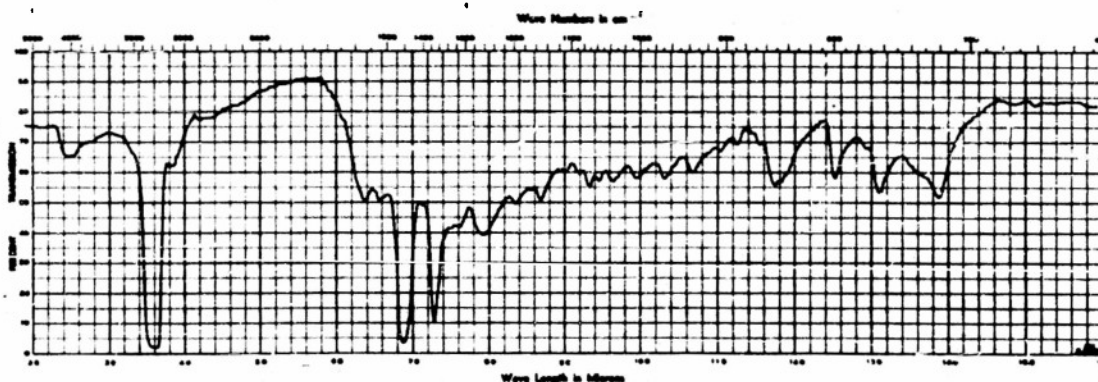
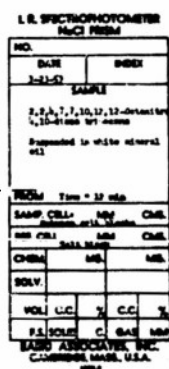


Ultraviolet Absorption Spectrum of 2,2,4,7,7,10,12,12-Octanitro-4,10-diazatridecane in Acetonitrile

20. Melting point: 206-208 °C.(d)

A blank graph grid for plotting temperature vs. time. The vertical axis is labeled "TEMPERATURE °C." and the horizontal axis is labeled "TIME". The grid is 10 units wide and 10 units high.

<u><0.1</u>	g/100 ml H ₂ O at 25°C.	<u> </u>	g/100 ml H ₂ O at <u> </u>	°C.
<u>1.4</u>	g/100 ml <u>acetone</u>		at <u>25</u>	°C.
	(name material used as solvent)			
<u><0.1</u>	g/100 ml <u>toluene</u>		at <u>25</u>	°C.
	(name material used as solvent)			



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the followings: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strins of cigarette paper or carbon paper and compress the stack in a C clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:

(a) By itself _____

(b) In mixtures (with additives) _____

(c) Inhibiting action on polymerization of:

Thiokol _____

Methacrylate _____

Other compounds _____

30. Availability

a. Amount now available? Research quantities _____

b. When was available material first prepared? _____

c. Amount prepared at that time? _____

d. Is large production feasible? _____

e. Plant capacity in existence, lbs/day? _____

f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 126

Calculated Ballistic Mortar Value = 149

Method of Aerojet Report No. 512, p. 6

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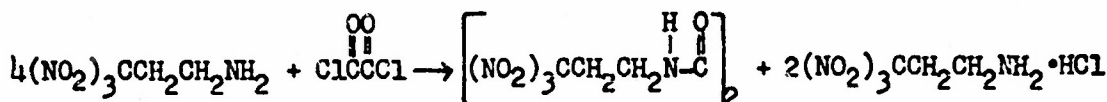
Tests Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound farther. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: N,N'-bis(3,3,3-Trinitropropyl)-

Name oxamideEmpirical formula C₈H₁₀N₈O₇Structure: (configuration) V

Information submitted by:

Activity Aerojet-General CorporationPerson M.B. Frankel and L.T. CarletonDate 1 August 1953Preparation reaction(s): V

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	21.73	2.28		25.34		
By determination	22.06	2.32		25.31		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test	Recommended method	
a. Impact Sensitivity	NOL OSRD 3401 p.8	2.5-kg weight
b. Thermal Stability	OSRD 3401 p.8	
c. Vacuum Stability	NOL OSRD 3401 p.8	48 hr at 100°C
d. Temperature of Explosion	OSRD 3401 p.8	
e. Temperature of Ignition	NOL OSRD 3401 p.8	
f. Impact Stability	Bureau of Mines, Bull. No. 346, p. 72 (2-kg weight)	
g.		
h.		

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetryl, N.C., etc.)

New Compound test results

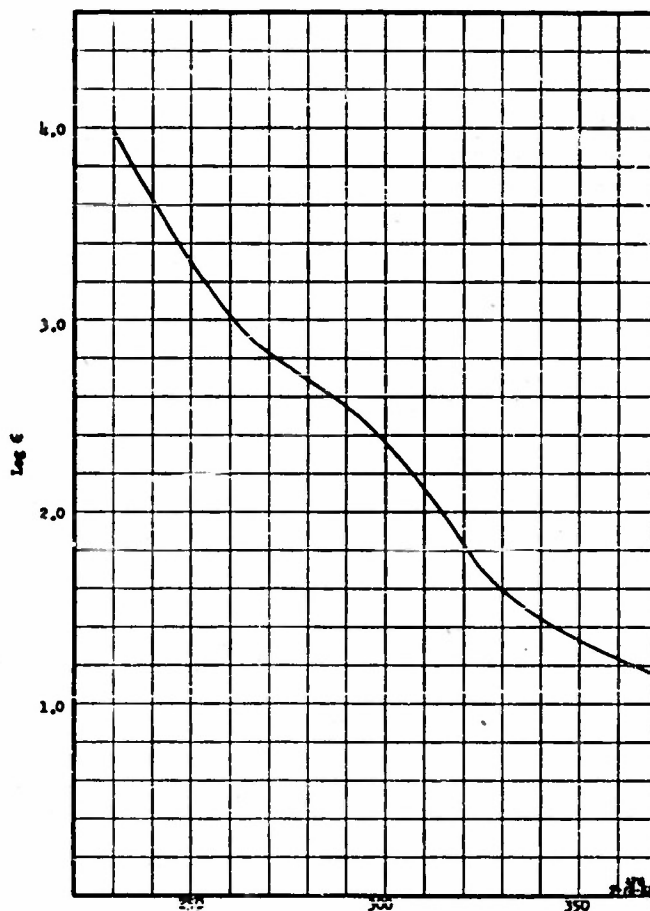
a. Tetryl, 32 cm/2.5 kg	45 cm/2.5 kg
b.	
c.	0.31 cc/g
d.	
e.	215°C
f. RDX, 30 cm/2 kg	15-20 cm/2 kg
g.	
h.	

4. Heat of formation: (ΔH) \pm -145 Kg. calories at 25°C., 1 atm. pressure
(indicate sign)

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Report No. 737
SPIA/443

	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2146	cal/gm	2175
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	Fine, needle-shaped crystals		
9. Simple microscope analysis data: (crystal studies)			
10. Density (Macro method)	1.692 gm/cm ³ (NOL)	(Micro or other method)	gm/cm ³ (Indicate on separate sheet any unique methods you use.)
11. Index of refraction (n _D ^{25°C.})		12. Color	White
		13. Odor	None
14. pH at 25°C.	5.6	(Method reference Q88D 1401 v.4, or Q88D 5968. Indicate method used, i.e. solvent and concentrations used. pH indicator water or Beckman pH meter.)	
	pH meter, 0.087 M in acetone/water (5/1 volume ratio)		



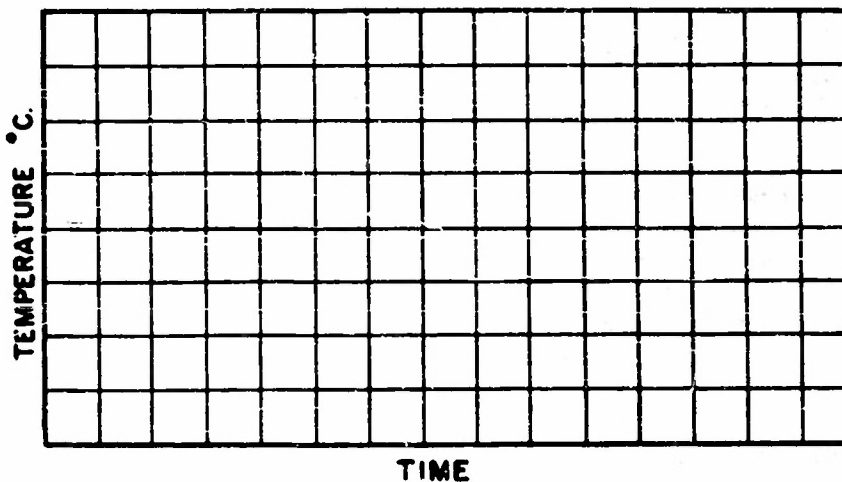
Ultraviolet Absorption Spectrum of N,N'-bis(3,3,3-Trinitropropyl) Oxamide in Ethanol

20. Melting point: 195-197 °C. (d)

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Report No. 737
SFA/MS

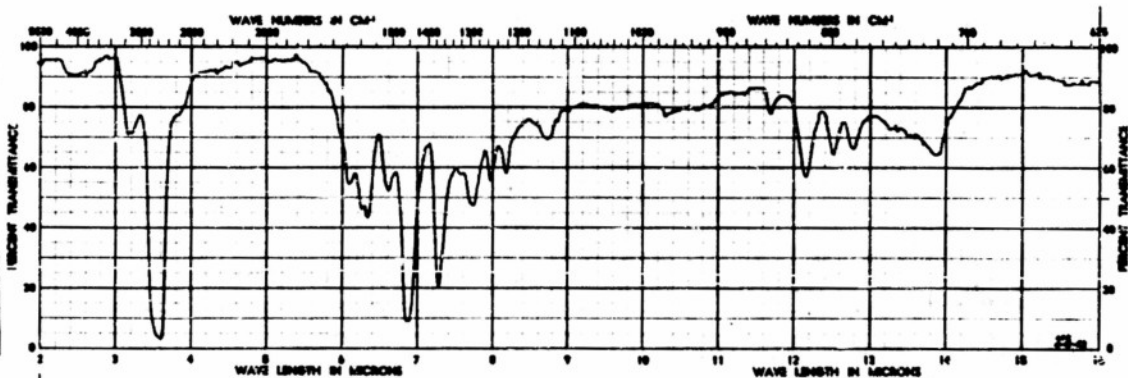
21. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



22. Solubility of new compound:

<0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
17 g/100 ml acetone at 25 °C.
 (name material used as solvent)
0.3 g/100 ml toluene at 25 °C.
 (name material used as solvent)

I.R. SPECTROMETER			
MCT PRISM			
NO.	DATE	INDEX	
5/5/50		TABLE	
N, N'-bis(3,3,3-trichloropropyl)			
diamide			
Dissolved in water at room temp.			
403			
FROM: Chem. 12. 10. 10.			
SAMP. CELL	MM	CM	
REF. CELL	MM	CM	
CHINA	MM	CM	
SOLV.			
VOL. C.C.	% C.C.	%	
10.00	10.00	10.00	
KIMMEL ASSOCIATES, INC.			
CAMDEN, N.J., U.S.A.			
1950			



Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____
30. Availability
a. Amount now available? Research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existence, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 135)
Calculated Ballistic-Mortar Value = 133) Method of Aerojet Report No. 512, p. 8

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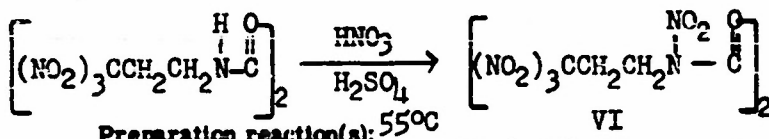
Data Questionnaire on

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COMPOUND: N,N'-Dinitro-N,N'-bis(3,3,3-trinitro-
Name propyl) oxamide
Empirical formula C₈H₈N₁₀O₁₈
Structure (configuration) VI

Information submitted by:

Activity Aerojet-General CorporationPerson M.B. Frankel and L.T. CarletonDate 1 August 1953

1. Quantitative analysis(% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	18.05	1.52		26.32		
By determination	18.42	1.63		26.49		

2. Burning properties:(compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test		Recommended method	
a. Impact Sensitivity	NOL	TEST METHOD	2.5-kg weight
b. Thermal Stability		OSRD 3401 p.8	
c. Vacuum Stability	NOL	TEST METHOD	48 hr at 100°C
d. Temperature of Explosion		OSRD 3401 p.6	
e. Temperature of Ignition	NOL	TEST METHOD	
f. Impact Stability	Bureau of Mines, Bull. No. 346, p. 72 (2-kg weight)		
g.			
h.			

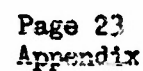
RESULTS OF ABOVE TESTS

Reference compound (designation-TNT, Tetryl, N.C., etc.)	New Compound test results
a. Tetryl. 32 cm/2.5 kg	9 cm/2.5 kg
b.	
c.	0.40 cc/g
d.	
e.	207
f. RDX. 30 cm/2 kg	10 cm/2 kg
g.	
h.	

4. Heat of formation: (ΔH) + -90 Kg. calories at 25°C., 1 atm. pressure
(Indicate sign)

A blank graph grid for plotting temperature vs. time. The vertical axis is labeled "TEMPERATURE °C." and the horizontal axis is labeled "TIME". The grid is 10 units wide and 10 units high.

<u>< 0.1</u>	<u>g/100 ml H₂O at 25°C.</u>	<u>g/100 ml H₂O at</u>	<u>°C.</u>
<u>89</u>	<u>g/100 ml acetone</u>	<u>at</u>	<u>25 °C.</u>
	<u>(name material used as solvent)</u>		
<u>7.0</u>	<u>g/100 ml toluene</u>	<u>at</u>	<u>25 °C.</u>
	<u>(name material used as solvent)</u>		



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Report No. 737
SFIA/M3

Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OSRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of exudation or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a C-clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____
30. Availability
a. Amount now available? _____ Research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existence, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 176
Method of Aerojet Report No. 512. p. 8
Calculated Ballistic-Mortar Value = 147

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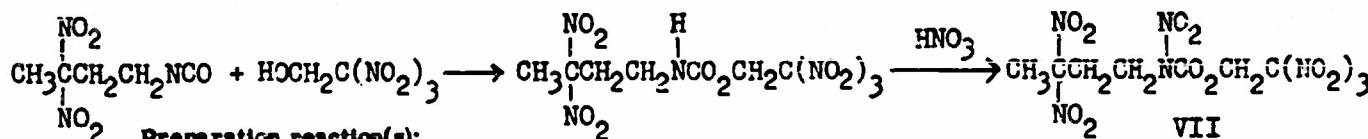
Data Questionnaire on
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

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COMPOUND: 1,1,1,5,8,8-Hexanitro-3-oxa-4-keto-

Name 5-aza-nonaneEmpirical formula C₇H₅N₆O₁₁Structure (configuration) VII

Information submitted by:

Activity Aerojet-General CorporationPerson M. B. Frankel and L. T. CarletonDate 1 August 1953

Preparation reaction(s): _____

1. Quantitative analysis (% by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	20.25	2.19		23.62		
By determination	20.25	2.21		23.67		

2. Burning properties: (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.) _____

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test		Recommended method	
a. Impact Sensitivity	NOL	XXXXXX	2.5-kg weight
b. Thermal Stability		OSRD 3401 p.6	
c. Vacuum Stability	NOL	XXXXXX	28 hr at 100°C
d. Temperature of Explosion		OSRD 3401 p.6	
e. Temperature of Ignition	NOL	XXXXXX	
f. _____		_____	_____
g. _____		_____	_____
h. _____		_____	_____

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetryl, H.C., etc.)

New Compound test results

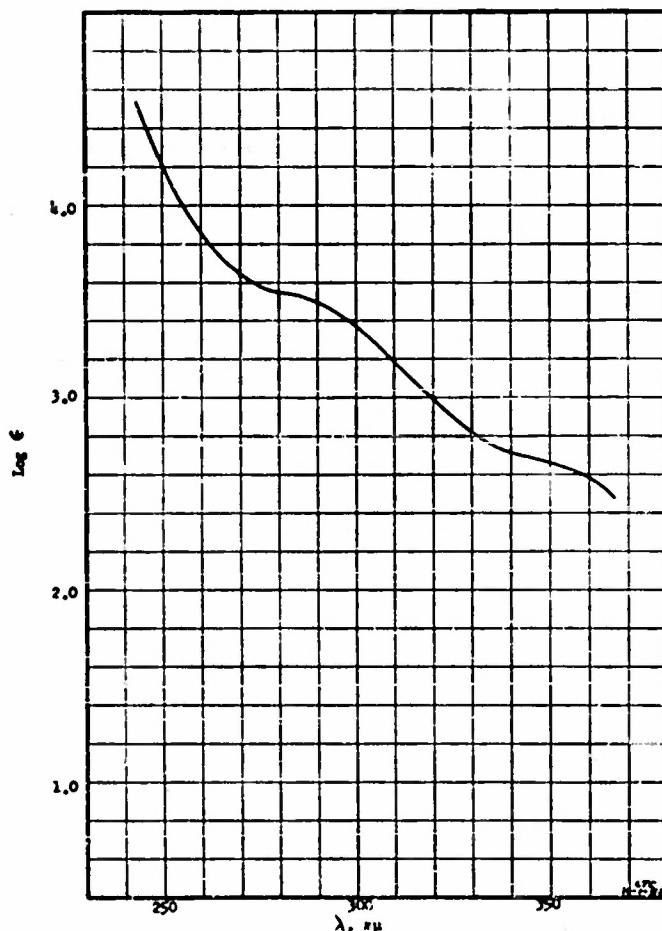
a. Tetryl. 32 cm/2.5 kg	15 cm/2.5 kg
b. _____	_____
c. _____	0.50 cc/g
d. _____	_____
e. _____	221°C
f. _____	_____
g. _____	_____
h. _____	_____

4. Heat of formation: (ΔH) + -132 Kcal. calories at 25°C., 1 atm. pressure
(Indicate sign)

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Report No. 737
SP1A/M3

	By Experiment	By Calculation	Method Description or reference. Separate sheet if necessary.
5. Energy of explosion (Q) (at 25°C. H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 25°C. H ₂ O liquid)	2007	cal/gm	2032 Aerojet Report No. 417A
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	Crystalline solid		
9. Simple microscope analysis data: _____ (crystal studies)			
10. Density (micro method)	1.733 gm/cm ³	(Micro or other method)	_____ gm/cm ³
(NOL) (Analyze on separate sheet any unique methods you use.)			
11. Index of refraction (n _D ²⁰)		12. Color	White 13. Odor None
14. pH at 25°C.	4.5	(Method reference OECD 3401 v.4, or OECD 5965. Indicate method used, i.e. solvent and concentrations used. pH indicator water or Beckman pH meter.) (decreasing rapidly)	
With Beckman pH meter. 0.0091 M in acetone/water (5/1 volume ratio)			



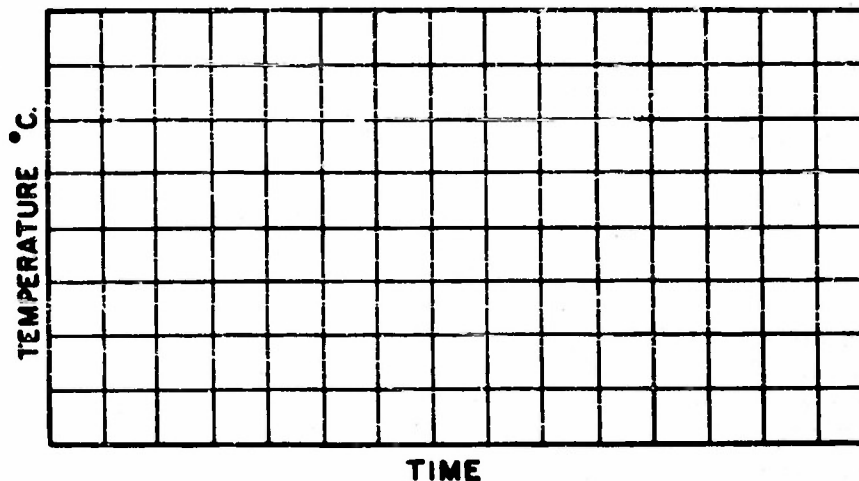
Ultraviolet Absorption Spectrum of 1,1,1,5,8,8-Hexanitro-3-oxa-4-azabicyclo[3.3.0]octane in Ethanol

20. Melting point: 107-108 °C.

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Report No. 737
SP1A/M3

31. Freezing point vs. Time Curve: (Please mark the coordinates with scale values that apply to the compound under study.)



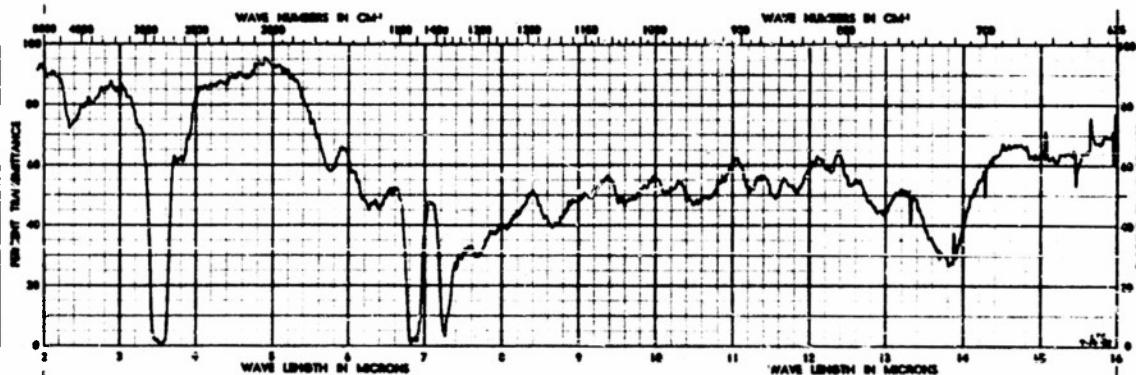
32. Solubility of new compound:

< 0.1 g/100 ml H₂O at 25°C. _____ g/100 ml H₂O at _____ °C.
140 g/100 ml acetone at 25 °C.
 (name material used as solvent)
14 g/100 ml toluene at 25 °C.
 (name material used as solvent)

I.R. SPECTROPHOTOMETER
Nujol Mixture

NO.	DATE	PREP.
7-34-52		
SAMPLE		
1,1,1,2,2,2-hexachloro-1,2-dichloroethane		
Dissolved in white mineral oil		
NECH Time = 12 min		
SAMP. CELL	MM	CHL.
REF. CELL	MM	CHL.
CHGR	MM	MM
SOLV.	MM	MM
VOL.	CC	CC
WAVELENGTH	CM	CM

SAFETY ASSOCIATES, INC.
CAMBRIDGE, MASS., U.S.A.



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Report No. 737
SPIA/MS

Under compatibility we are considering the ability of two compounds to be in intimate contact (Note OQRD 5758 p. 21-22) over a long period of time without adverse effects on either the chemical or physical properties of either material. These tests will probably be of varied extent. They might be some of the following: (a) Standard stability tests. (b) Simple observations of emulsion or separation at ambient or accelerated temperature. (c) Prepare thin sheets (.025" thick, 1" square) of the plasticized material. Separate the sheets with strips of cigarette paper or carbon paper and compress the stack in a "C" clamp. After several days note the oily collection on the paper. Please give reference to or describe procedure used. Item 28 is to indicate results when the compound is in contact with some material other than ethyl cellulose, nitrocellulose or rubber.

25. Compatibility with ethyl cellulose: _____

26. Compatibility with nitrocellulose: _____

27. Compatibility with rubber: _____

28. Compatibility with _____: _____

29. Polymerizing properties of the new compound:
(a) By itself _____
(b) In mixtures (with additives) _____
(c) Inhibiting action on polymerization of:
 Thiokol _____
 Methacrylate _____
 Other compounds _____
30. Availability
a. Amount now available? Research quantities
b. When was available material first prepared? _____
c. Amount prepared at that time? _____
d. Is large production feasible? _____
e. Plant capacity in existence, lbs/day? _____
f. Outline steps for a quantity production method _____

31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 153
Calculated Ballistic-Mortar Value = 140 Method of Aerojet Report No. 512, p. 8

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Data Questionnaire on

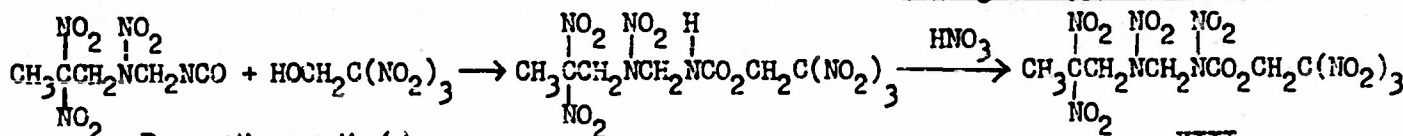
COMPOUNDS FOR USE AS INGREDIENTS OF PROPELLANTS AND OTHER EXPLOSIVES

Listed below are the characteristics which are believed to be of importance in the study of a compound which may possibly be used as a constituent of solid propellants or other explosives. It is not expected that all this information will be available for every compound. For example, with a compound showing poor stability, it is probably not advisable to investigate that compound further. After placing such information as is available for a compound which you have prepared or tested on one of these forms, send it to SOLID PROPELLANT INFORMATION AGENCY, AFL/JHU, 8621 Georgia Avenue, Silver Spring, Maryland. As additional information on the same or new compounds accrues, forward it on a similar form at a later date. The information submitted on these forms will be rewritten and published by SPIA in loose-leaf manual form. These forms may also be used as work or data sheets for your experimental studies. Extra copies are available upon request from SPIA. Suggestions for improvement of these forms are invited. If insufficient space has been provided for any item, attach separate sheets.

COMPOUND: 1,1,1,5,7,9,9-Heptanitro-3-oxa-4-keto-

Name 5,7-diaza-decaneEmpirical formula C₇H₅N₇O₇Structure (configuration) VIII

Information submitted by:

Activity Aerojet-General CorporationPerson M.R. Frankel and L.T. CarletonDate 1 August 1953

Preparation reaction(s):

VIII

1. Quantitative analysis (by weight)

	Carbon	Hydrogen	Oxygen	Nitrogen		
Calculated from formula	17.69	1.91		26.53		
By determination	18.08	1.95		26.58		

2. Burning properties (compared to nitrocellulose, under nitrogen at atmospheric pressure:)

(faster? slower? residue? etc.)

3. Stability and Sensitivity: Plot any graphs on separate sheet

Discuss methods used when they vary from references. Give temperature used. (Use separate sheet if necessary.)

Name of test		Recommended method	
a. Impact Sensitivity	NOL	OSRD 3401 p.8	2.5-kg weight
b. Thermal Stability		OSRD 3401 p.8	
c. Vacuum Stability	NOL	OSRD 3401 p.8	18 hr at 100°C
d. Temperature of Explosion		OSRD 3401 p.8	
e. Temperature of Ignition	NOL	OSRD 3401 p.8	
f.			
g.			
h.			

RESULTS OF ABOVE TESTS

Reference compound

(designation-TNT, Tetryl, N.C., etc.)

New Compound test results

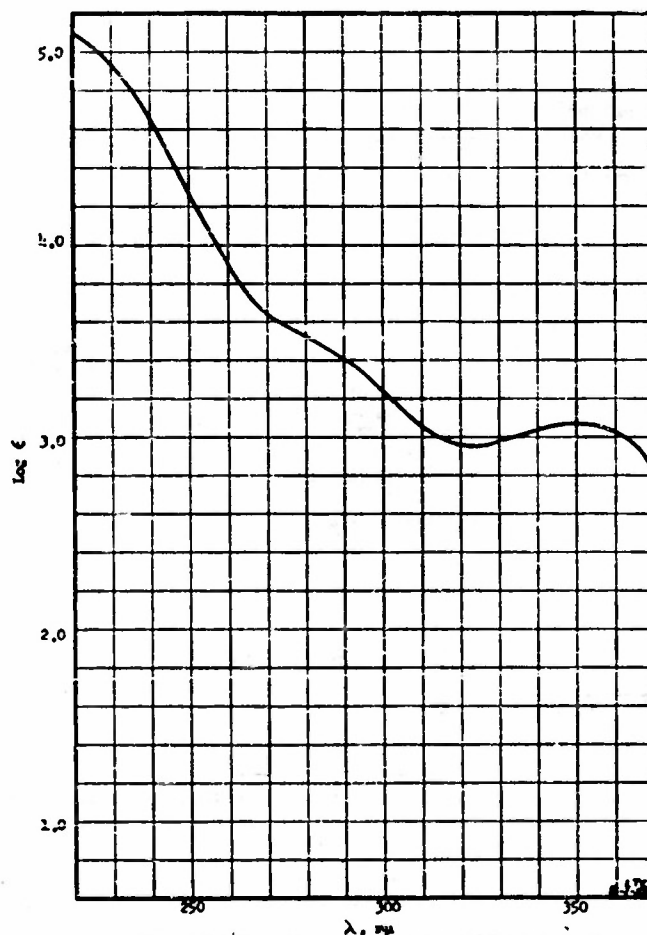
a. Tetryl, 32 cm/2.5 kg	18 cm/2.5 kg
b.	
c.	1.01 cc/g
d.	
e.	219°C
f.	
g.	
h.	

4. Heat of formation: (ΔH) + $\frac{-118}{(\text{indicate sign})}$ Kg. calories at 25°C., 1 atm. pressure

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SPLA/MS

	By Experiment	By Calculation	Method <small>Description or reference. Separate sheet if necessary.</small>
5. Energy of explosion (Q) (at 2500, H ₂ O liquid)		cal/gm	
6. Heat of combustion (H _c) (at 2500, H ₂ O liquid)	1785	cal/gm	1798
7. Specific impulse (I _{sp}) calc:		lb-sec/lb	
8. Physical form of compound (viscous liquid, crystalline type, etc.)	Flaky crystals		
9. Simple microscope analysis data: _____ (crystal studies)			
10. Density (Macro method)	1.790 gm/cm ³	(Micro or other method)	gm/cm ³
(NOL) (Krolein on separate sheet any unique methods you use.)			
11. Index of refraction (n _D ^{25°C})		12. Color	White
		13. Odor	None
14. pH at 25°C.	3.5	(Method reference OSRD 3401 v.4, or OSRD 5966. Indicate method used, i.e. solvent and concentrations used. pH indicator used or Beckman pH meter.) (decreasing rapidly)	
With Beckman pH meter, 0.0091 M in acetone/water (5/1 volume ratio)			



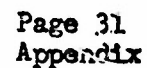
Ultraviolet Absorption Spectrum of 1,1,1,5,7,9-Heptanitro-4-oxo-5,7-dioxo-decane in Methylene

20. Melting point: 140-141 °C.

TEMPERATURE °C.

TIME

<u>0.1</u>	g/100 ml H_2O at <u>25</u> °C.	<u> </u>	g/100 ml H_2O at <u> </u> °C.
<u>76</u>	g/100 ml <u>acetone</u>	at	<u>25</u> °C.
	(name material used as solvent)		
<u>0.1</u>	g/100 ml <u>toluene</u>	at	<u>25</u> °C.
	(name material used as solvent)		



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31. Additional information: (toxicity, hazards, deterioration, oxygen balance, detonation rate, explosive power, ease of hydrolysis by water, etc. List references, reports, data books, etc. that refer to the compound.) _____

Calculated Lead-Block Value = 167 }
Calculated Ballistic-Mortar Value = 104 } Method of Aerojet Report No. 512, p. 8

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N7onn-462, Task Order I, NOaz 53-618-c

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